# AEC/NASA SYMPOSIUM

# on

# CONTAMINATION CONTROL

## Current and Advanced Concepts in Instrumentation and Automation

Held at

HOLIDAY INN
Albuquerque, New Mexico

September 12-14, 1967

Issued by Sandia Corporation,
a prime contractor to the
United States Atomic Energy Commission

PRECEDING PAGE BLANK NOT FILMED.

TABLE OF CONTENTS

TABLE OF CONTENTS (cont)

TABLE OF CONTENTS (cont)

ATTENDANCE

Chairman - H. D. Sivinski, Sandia Corporation
Vice Chairman - F. J. Beyerle, Marshall Space Flight Center

SANDIA CORPORATION
Albuquerque, New Mexico

Aubuchon, D. W.
Ballard, D. W.
Baxter, H. H.
Burchett, O. L.
Champe, C. E.
Clement, W. L.
Davis, D. M.
DeLollis, N. J.
Dillon, R. T.
Drake, E. L.
Dunaway, E. R.
Fjelseth, D. F.
Garst, D. M.
Gruer, E. R.
Guess, T. R.
Henderson, R. W.
Hey, N. S.
Holton, J. D.
Hornbeck, J. A.
Jennings, C. W.
Kenagy, J. A.
Klamerus, L. J.
Krieger, G. L.
Lenander, H. E.
Lenert, G. B.
Lindell, K. F.
Mashburn, J. C.
Mills, C. R.
Morris, M. E.
Neitzel, W. E.
Oswalt, F. W.
Page, C. L.
Perryman, R. E.
Quant, A. J.
Reynolds, M. C.
Roper, L. C.
Roth, E. S.
Runyan, C. E.
Rush, E. E.
Schreiner, E. T.
Shepherd, E. W.
Sivinski, H. D.
Smith, S. L.
Trauth, C. A.
Utter, R. F.
Van Domelen, B. H.
Whitfield, W. J.

ATOMIC ENERGY COMMISSION
Washington, D. C.

Gilbert, H.

NASA
Washington, D. C.

Negola, D. E.
Neill, A. H.

NASA/WHITE SANDS TEST FACILITY
Las Cruces, New Mexico

Peipelman, B. W.
Smith, I. D.

MARSHALL SPACE FLIGHT CENTER
Huntsville, Alabama

Beyerle, F. J.
Rush, C. M.
Vickers, D. N.

AMES RESEARCH CENTER
Sunnyvale, California

Burgard, J. P.

KENNEDY SPACE CENTER
Cape Kennedy, Florida

Gayle, J. B.
Green, E. L.
Hull, L. C.

NASA
Cocoa, Florida

Petty, W. F.

GODDARD SPACE FLIGHT CENTER
Greenbelt, Maryland

LeDoux, F. N.

ATTENDANCE

LEWIS RESEARCH CENTER
Cleveland, Ohio

Adams, D. C.

MANNED SPACE CENTER
Houston, Texas

Ussery, Q. T.

UNIVERSITY OF NEW MEXICO
Albuquerque, New Mexico

Beakley, J. W.
Franklin, C.
Hughes, L. W.

LOVELACE CLINIC
Albuquerque, New Mexico

Schwichtenberg, A. H.
Whitcomb, J. G.

CONTAMINATION CONTROL JOURNAL
Reporter
Albuquerque, New Mexico

Kutnewsky, F.

ENVIRCO, INC.
Albuquerque, New Mexico

Marsh, R. C.

PALL CORPORATION
Albuquerque, New Mexico

Poage, L. O.

JET ELECTRONICS CORPORATION
Albuquerque, New Mexico

Birrell, G. R.
Smith, D. W.
Tillman, J. E.

ST. VINCENT'S HOSPITAL
Santa Fe, New Mexico

Goodrich, E. O.

AEROJET GENERAL CORPORATION
Las Cruces, New Mexico

Mountain, W. E.

GRUMMAN AIRCRAFT CORPORATION
Las Cruces, New Mexico

Hicks, J. A.
Seib, B. G.

GRUMMAN AIRCRAFT CORPORATION
Bethpage, New York

Kolodzeski, L. J.
Marinaro, D. S.
Marx, H. J.

HAYES INTERNATION CORPORATION
Birmingham, Alabama

Ellenburg, J. Y., Mrs.

U. S. PUBLIC HEALTH SERVICE
Phoenix, Arizona

Bond, W. W.
Favero, M. S.
Petersen, N. J.

NORTH AMERICAN AVIATION
Anaheim, California

Allison, F. M.

HIGH ACCURACY PRODUCTS CORPORATION
Claremont, California

Carver, L. D.

ATTENDANCE

FARR COMPANY
El Segundo, California

Lebadie, P. A.

MINES SAFETY APPLIANCE
Los Angeles, California

Becker, J. W.

MINE SAFETY APPLIANCES
Pittsburgh, Pennsylvania

Elinsky, H. C.

ROYCO INSTRUMENTS, INC.
Manhattan Beach, California

Theis, J.
Weersing, J. R.

ROYCO INSTRUMENTS, INC.
Menlo Park, California

Ogle, H. M.

JET PROPULSION LABORATORY
Pasadena, California

Hobby, G. L.

CONSULTANT
San Diego, California

Paulhamus, J. A.

MARTIN-MARIETTA COMPANY
Denver, Colorado

Cole, J. E.
McDonald, J. P.
Piccone, M.

BENDIX CORPORATION
Cape Kennedy, Florida

Briles, O. M.
Killion, J.
Underwood, W. F.

BENDIX CORPORATION
Kansas City, Missouri

Foster, R. T.
Hedrick, R. B.

GENERAL ELECTRIC
Daytona Beach, Florida

Conley, D. L.

GENERAL ELECTRIC
Philadelphia, Pennsylvania

Gillis, J. R.

GENERAL ELECTRIC
Pottstown, Pennsylvania

Gergle, J. J.

HEATING, PIPING, AIR CONDITIONING
Chicago, Illinois

Elliott, T. C.

IIT RESEARCH INSTITUTE
Chicago, Illinois

Jackson, M. R.
Lieberman, A.
Saperstein, Z. P.

IOWA STATE UNIVERSITY
Ames, Iowa

Voss, M. D.

ATTENDANCE

MASON AND HANGER
Burlington, Iowa

Polson, J. R.

MASON AND HANGER
Amarillo, Texas

Burling, G. H.

THE BOEING COMPANY
New Orleans, Louisiana

Schonhard, G. P.

CHRYSLER CORPORATION
New Orleans, Louisiana

Hildebrandt, N. E.

AIR TECH
Baltimore, Maryland

Steinberg, S. B.

FAIRCHILD HILLER CORPORATION
Germantown, Maryland

Ross, A. P.

BARNSTEAD STILL & STERILIZER CO.
West Roxbury, Massachusetts

Smith, V. C.

UNIVERSITY OF MINNESOTA
Ph.D. Candidate
Minneapolis, Minnesota

Fey, F. L.

BELL TELEPHONE LABORATORIES
Murray Hill, New Jersey

Brown, R.
Cleveland, H. M.
Helmke, G. E.

WESTERN ELECTRIC
Princeton, New Jersey

Altman, C.
Shaw, E. J.

WESTERN ELECTRIC
Allentown, Pennsylvania

Morrison, P. W.

WESTERN ELECTRIC
Reading, Pennsylvania

Yeich, R. A.

KOLLSMAN INSTRUMENT COMPANY
Syosset, New York

Ravitz, P. I.

CAMBRIDGE FILTER CORPORATION
Syracuse, New York

Goulet, R. T.

FLANDERS FILTERS, INC.
Riverhead, New York

Cadwell, G. H.

BAUSCH AND LOMB INC.
Rochester, New York

Martens, A. E.

BATTELLE MEMORIAL INSTITUTE
Columbus, Ohio

Gieseke, J. A.
Pilcher, J. M.

NATIONAL BUREAU OF STANDARDS
Washington, D. C.

Paulson, R. A.

Edited and Prepared by MARIO J. SANGUINETTI
Sandia Corporation
Albuquerque, New Mexico

# OPENING

## Presentations

1. WELCOMING REMARKS -- J. A. Hornbeck
2. INTRODUCTION -- R. W. Henderson

Moderator

H. D. SIVINSKI
Sandia Corporation
Albuquerque, New Mexico

# WELCOMING REMARKS

by

JOHN A. HORNBECK
President
Sandia Corporation
Albuquerque, New Mexico

It is certainly a pleasure for me to be here this morning and to represent the Atomic Energy Commission and Sandia in welcoming all of you to the Contamination Control Symposium. For those of you who are unacquainted with the local situation here, Sandia is a subsidiary of the Western Electric Company, but is a prime contractor to the Atomic Energy Commission. In fact, Sandia has only one contract and that is with the AEC. All of the business it does is for the AEC, or on occasion for the Department of Defense through the AEC. Thus, although this is a joint AEC-NASA symposium, I feel that I can welcome you for the AEC.

But I also feel that, at least historically, I can welcome you on behalf of the National Aeronatic and Space Administration, since I spent four and a half years with Bellcomm before coming to Sandia. Bellcomm is a Bell System organization that also had only one contract and that was with NASA. There we were primarily engaged in systems engineering on the Apollo Program for landing men on the moon in this decade. With that one step removed, I would like to welcome you here on behalf of NASA also.

The third way I should welcome you is as a good resident of the sun country of the southwest, on behalf of Albuquerque and the State of New Mexico. Just a few miles from here there is a tramway that goes up to the top of the Sandia crest. It has only been in operation for less than two years, and it is the longest tramway in the United States. It provides a lovely trip up to the top.

I have just a few remarks that I would like to make on contamination control. These are somewhat personal, and my viewpoint will not be that of each of you, but I would like to call to your attention what may be rather obvious--the duality of the contamination control subject. In one sense, the purpose is clearly the control of contamination, but in another sense contamination control is involved in very much the same thing as the observation of new physical processes. Sir Isaac Newton was involved in an aspect of contamination control when an apple fell on his head: he was, in a sense, contaminated. The incident called his attention, however, to a physical process, namely that of gravitation. An extraneous event occurred that led him to a very fundamental observation.

In my own experience, some years ago, the duality of contamination control was a very real part of the everyday world in which I lived. In 1953, the first single crystals of silicon were made at Bell Telephone Laboratories in New Jersey. Transistors and other semiconductors are so well known now and so widely used that to some of you it may seem strange that there was a time when we had difficulty in making a single crystal of silicon. One aspect of contamination control in semiconductors concerns the group 3 and group 5 impurities which control the resistivity of the material. This was recognized at the time. However, a colleague and I decided to look into another aspect of the properties of silicon, and this had to do with its photoconductivity: if some light were shone on a single crystal, a conductivity change would occur in the material, and this increased conductivity could persist for some time. Actually, the study of photoconductivity in this kind of effect was quite old. Well known research people in Germany had studied the phenomena in the early 1900's and into the 1920's, when long-term photoconductivity effects in various materials were a great deal more mysterious than they are today. The photoconductivity effect in silicon we pinned down to be associated with traps for electrons or holes, that is for the minority carriers in the silicon material. In one type of silicon material, we found three traps for minority carriers. With three sets of traps we were able to account for all the photoconductivity effects lasting from fractions of a microsecond to tens of thousands of seconds. We had a theoretical model which explained what was going on.

The duality here is quite clear. The traps are associated with impurities in the material, in the single crystal of silicon, and the physical effect, which is the other way of looking at it, is the photoconductivity. The same kind of observation can be made with respect to color centers in alkali halide crystals. When an atom is missing from the lattice and an electron happens to be trapped in that site, we have one kind of color center and we have a defect or a contamination, but if there are interstitial atoms in the lattice, we have another kind of color center. And again we have the duality of an imperfection or contamination and a physical process.

These remarks lead me to one further observation--in pursuing the control of contamination we must have means for testing or detecting the contamination. We need measuring techniques for sensing as well as techniques for identifying the particular contamination. In the examples I gave, the color centers in alkali halide crystals, I identified an electron in an atomless lattice position as associated with the contamination. In the case of the normal conductivity of semiconductors, the contaminant was associated with the amount of group 3 or group 5 impurities in the lattice. We can not only identify the contaminant but also measure the amount of it present. With photoconductivity we knew the traps were present and we could measure the number, but at that time we could not identify what material was added to the silicon which was itself the cause of the traps. So in one case we do identify and in the other case we do not. It is important, for obvious reasons, both to sense and to identify.

It is clear from these remarks that I am not making an address. My principal objective in being here is to welcome you to Albuquerque and to encourage you, if you have more time, to stay beyond the Contamination Control Symposium and enjoy the Land of Enchantment. Thank you.

# INTRODUCTION

by

R. W. HENDERSON
Vice President
Sandia Corporation
Albuquerque, New Mexico

Over the past few years, there has been a tremendous increase of attention paid in laboratories, development groups, manufacturing concerns, and medical facilities to the extreme importance contamination control plays in achieving desired goals in the accomplishment of the task at hand. In all too many cases, the failure to achieve these goals has been traced to inadequate contamination control. Failure to succeed at any task costs dollars in scrappage, rework, doing the job over again, or resorting to an alternate, less attractive solution to the problem to avoid the difficulty. The pressure of competition for available dollars demands that every possible step be taken to do the job right the first time, and that goal has generated intensive work by many to develop techniques and controls that will assure step by step maintenance of acceptable contamination levels.

Contamination has many things in common with death and taxes. Left alone and ignored in this world's exponentially expanding activities, it will behave exactly like entropy -- increase, ad infinitum. But, as in death and taxes, organized probing attacks by dedicated competent people can at least deter its voracious effects, and, hopefully, eliminate them in some areas of prime importance. Knowing the nature of a contaminant, its origin, and how it reacts with or responds to attacking agents or mechanisms is half the battle in achieving its elimination or preventing its infiltration at the outset.

To be sure, control of contamination has been considered something of a virtue since mankind became aware of its existence, but, all too often, it was tackled as an afterthought, thereby resulting in cobbled-up appendages to the activity, product, or process in question. Beneficial results thus achieved are characteristically costly and may even result in degradation of the effectiveness of the activity, product, or process that the contamination control effort was supposed to improve.

Obviously, therefore, there was and is major need for consideration of contamination control as an integral part of the initial inputs to design requirements so that evaluated trade-offs can be made continuously by manipulations of these many requirements throughout the evolution of the design. Only by this give and take mechanism can one arrive at an integrated solution approaching the optimum.

An excellent illustration, being kept in front of the public at this time through national news media, is the attack upon objectionable constituents in the emissions from the internal combustion engines of motor vehicles. The first top-of-the-head ideas on control of this problem rather understandably focused upon exhaust scrubbers of one type or another, and numerous untrained, though vocal self-styled automotive engineers, rose up from the lay public with ready add-on "solutions." Simultaneously the laboratories of the automotive industry started to analyze the content of the total emissions to identify specific chemical components and their concentration.

The first source of emission of unburned hydrocarbons tackled was blow-by past the pistons, into the crankcase, and out the crankcase breather pipe to the atmosphere--a large contribution, particularly in older cars with worn piston rings and cylinders. A 30 percent reduction in total emission was achieved on 1961 models for sale in California, and for nationwide sale on 1963 models by the addition of a feed-back path from the crankcase to the carburetor outlet to the engine, thereby eliminating the crankcase breather.

Since proper air-to-fuel ratio is essential to efficient combustion, complicated by this crankcase vapor return, it was found that the daily and annual wide variations in air temperature had to be grossly reduced. This is being done on 1968 models through the incorporation of an air preheater which uses the heat from the exhaust manifold. Thus the air intake to the carburetor will be temperature controlled to a much greater degree, permitting better control of the air to fuel ratio over the daily driving cycle.

In addition to these two major improvements, air is to be injected into the hot exhaust manifold to accomplish more complete combustion of the unburned hydrocarbons and the conversion of carbon monoxide to carbon dioxide as they leave the cylinders. The additional heat generated in this process will result in a hotter exhaust system with its consequent design and materials compatibility problems in mufflers--a fine example of the need for integrated design from start to finish wherein all factors are considered simultaneously.

These three improvements in design will result in a 60 percent reduction in objectionable emissions; but with that in hand, another significant source comes into focus--the surprising amount of gasoline that evaporates directly to the atmosphere from the hot carburetor assembly and from the gasoline tank breather system. These two sources are quite appreciable, so work is progressing on their elimination. Lower vapor pressure fuels would help, but their use would reflect directly back into carburetor and engine design if current levels of horsepower per pound of engine weight are to be maintained.

And so it goes. The elimination or control of major sources of contamination uncovers also-rans that emerge into the spotlight for similar attack--a never ending challenge that the world can no longer blithely ignore if its health and future well-being are to be guaranteed.

Since much of the work to date and hoped for in the future to eliminate this contamination control problem has been or will be accomplished through the expenditure of taxpayers' dollars, directly or indirectly, we are all obligated to share our successes, and often equally important failures, with each other and with society at large, to the end that the wheel will not be repeatedly reinvented and maximum progress can be achieved at the earliest point in time and at the lowest dollar cost.

Therefore, the objectives of this Symposium are:

1. To provide a means of exchanging technological information concerning advances in automation and advanced instrumentation for contamination control.

2. To provide a better understanding of capabilities and limitations of contamination control monitoring equipment.

3. To explore future needs for contamination control instrumentation.

To address these objectives, the program has been arranged into six sessions:

Session 1. Contamination in liquids.

Session 2. Radiation monitoring.

Session 3. Surface contamination.

Session 4. Air and Gas contamination.

Session 5. Microbial contamination.

Session 6. Systems approach to contamination control.

Again we are pleased that so many of you have shown enough interest in the subject to travel many miles to exchange ideas and become better acquainted with others facing similar problems. We also consider it a distinct honor that this symposium is being held in Albuquerque. We hope that you will enjoy your stay in the Land of Enchantment and carry away with you more insight than you brought to this discussion of one of the greatest threats facing this country today, particularly as it relates to our atmosphere and water resources.

SESSION I

CONTAMINATION IN LIQUIDS

Presentations

| | | |
|---|---|---|
| 1. | NONVOLATILE RESIDUE TEST METHODS | -- D. N. Vickers |
| 2. | AN AUTOMATIC METHOD FOR MEASURING THE NONVOLATILE RESIDUE OF CLEANING SOLVENTS | -- M. R. Jackson |
| 3. | THE SOLVENT PURITY METER AND ITS APPLICATIONS TO PRECISION CLEANING | -- F. W. Oswalt |
| 4. | CONTAMINATION SENSORS | -- M. Piccone |
| 5. | MEASUREMENT OF CONDUCTIVITY OF LIQUIDS AS A MONITOR FOR CONTAMINATION | -- V. C. Smith |
| 6. | CONTAMINATION EFFECTS AND CONTROLS IN SATURN LAUNCH VEHICLE HYDRAULIC SYSTEMS | -- F. J. Beyerle |

Session Chairman

J. P. GAYLE
Kennedy Space Flight Center
Cape Kennedy, Florida

# SESSION I

# CONTAMINATION IN LIQUIDS

## INTRODUCTION

by

J. N. GAYLE

Kennedy Space Flight Center
Cape Kennedy, Florida

In the space industry, contamination control first gained a foothold as a separate discipline in connection with liquid systems when, in the mid-1950's, a small laboratory was established at Redstone Arsenal to sample and analyze hydraulic fluid for particulate content. Since then, the scope of contamination control has been enlarged to include almost every type of contaminant and media imaginable.

At Kennedy Space Flight Center, the importance of contamination control in liquid systems is reflected in the 1000 commodity analyses performed per week in support of the APOLLO program, with most of these commodities being liquids. In addition, the Air Force laboratory performs about 500 analyses per week in support of the unmanned launch programs.

The current rejection rate is on the order of 20 percent. Every time a sample is rejected, the impact may range from one extreme to the other. The minimum impact is going out and getting another sample. Or it may be a little more drastic in that the commodity has to be returned to the vender. And the maximum impact, of course, is the delay of a major test or launch. So it can be seen that there are real problems in contamination in liquid systems.

The first three papers in Session I are related. They all deal with the development of automatic instruments for the measurements of nonvolatile residue (NVR) content of solids. The practical importance of this particular measurement can again be illustrated by the operations at Kennedy Space Flight Center, where NASA and the Air Force together perform about 400 NVR determinations per week in support primarily of the various cleaning functions. Not only would a short, fast, and accurate method represent a significant savings in terms of manpower, but also it would represent a major savings in turn-around time for hardware, which is frequently idle since it cannot be packaged or put into the system while the results of the analyses are being awaited.

The last three papers of this session will deal with the more general problem of sampling and anslysis of fluids for the various contaminants, ranging from the overall problem involved in automating the monitoring of contamination in fluid systems, and the use of resistance measuring techniques to detect the presence of minute quantities of metallic contamination in certain solvents, to the effect of particulate contaminants on various hydraulic system components.

# 1. NONVOLATILE RESIDUE TEST METHODS

by

D. N. VICKERS

Marshall Space Flight Center
Huntsville, Alabama

Early in the space program it was discovered that most organic materials and liquid oxygen (LOX) make very sensitive and hazardous mixtures. This and other contamination factors resulted in some strict space hardware cleanliness requirements. The most frequently used oil cleanliness evaluation method in industry has been the water break test, which is more a "go, no go" test than a quantitative test. The nonvolatile residue (NVR), gravimetric test presently used was devised in an effort to obtain a quantitative answer; however, it was soon apparent that the method had many deficiencies as follows:

1. Evaporation, weighing, and reweighing of the sample is time consuming, especially in the case of high boiling solvents such as trichloroethylene. Trichloroethylene requires about 3 hours from start to finish for a single sample; however, on a production basis with the use of a flash evaporator, the average time per sample is less than 1 hour.

2. Part of the NVR is lost with solvent vapors during solvent evaporation, and this loss is not constant enough to allow the use of a correction factor.

3. Even if the analysis method were completely accurate, the results would still be only an approximation of actual surface contamination. Tests have shown that only a portion of contamination on a surface can be removed by test solvent flushing, and this portion is variable, depending upon flushing conditions.

Figure 1 shows typical NVR test rsults by the gravimetric method. Data are from analysis results for prepared samples of heavy mineral oil in trichloroethylene. Note that the percent recovery ranges from about 48 to 72 percent, and that percent recovery is not reproducible for a given contamination level, as evidenced by identical samples 1-2, and 3-5. These analyses were conducted at a controlled ambient temperature, about 33°C, to duplicate test conditions as closely as possible and to lose a minimum of oil by evaporation.

| SAMPLE | MILLIGRAMS OF OIL ADDED | MILLIGRAMS OF OIL RECOVERED | PERCENT RECOVERY |
|---|---|---|---|
| 1 | 5 | 3.12 | 62.4 |
| 2 | 5 | 2.40 | 48.0 |
| 3 | 10 | 7.22 | 72.2 |
| 4 | 20 | 15.12 | 75.6 |
| 5 | 10 | 5.45 | 54.5 |

NOTE: DATA FROM HAYES INTERNATIONAL CORPORATION, NASA TEST REPORT NO. 10911, VOLUME 1, CLEANING STUDY PROGRAM, CONTRACT NO. NAS8-2483.

Figure 1. Ambient temperature evaporation of trichloroethylene

To resolve these and other problems, help was solicited from industry, and in 1962 a contract was awarded to Illinois Institute of Technology Research Institute (IIT) for development of analytical methods and test equipment for determination of hydrocarbon contamination. The testing and analysis methods shown on Table I were studied.

TABLE I

Teating and Analysis Methods Studied by IITRI

1. On site-testing of launch vehicle surface cleanliness.
   - A. flash photolysis
   - B. adsorption - desorption
   - C. vacuum desorption
   - D. vibrating capacitor

2. Determining minute hydrocarbon contamination levels in chlorinated (trichloroethylene) flushing solvents.
   - A. attenuated total reflectance
   - B. critical angle ellipsometry
   - C. near-infrared spectroscopy
   - D. light scattering

Part 1 on the table lists surface cleanliness evaluation methods alternate to the solvent flush and NVR analysis method, which were evaluated, and they will not be discussed. Of the NVR analysis methods in Part 2 and several other methods which have been tried during the past 10 years, all but one were found to lack the required sensitivity in the range of 0 to 20 mg of oil per 500 ml of solvent. Light scattering was the only method tested which gave the desired accuracy and speed of analysis. The method measures NVR as a function of the amount of light scattered by the NVR droplets which are formed when solvent is evaporated from a filtered contaminated solvent aerosol.

Figure 2 presents a good picture of the sensitivity of the laboratory instrument used by IIT in its study of the light scattering method with Harmony 69 oil used as the contaminant. Note that the curve shows good sensitivity in the range of 0 to 20 ppm. The sensitivity was less for halogenated test contaminants such as KEL-F-10.

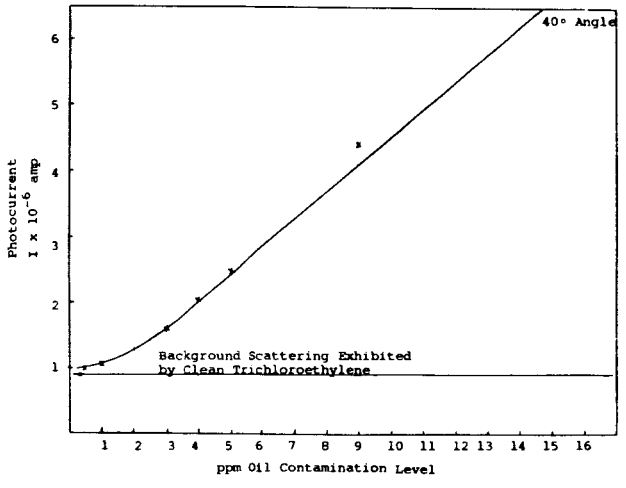



Figure 2. Total light scattering for Harmony 69 oil when diluted from 50 ppm standard solution to various contamination levels

As a result of this feasibility study, IIT furnished two prototype production instruments to the Marshall Space Flight Center for evaluation. The first instrument was evaluated by the Propulsion and Vehicle Engineering Laboratory, and its conclusion was that the method is faster and more accurate than the gravimetric method, but that the equipment could be improved. The second instrument incorporated improvements made as a result of the first instrument evaluation, and it is being evaluated by the Quality and Reliability Assurance Laboratory.

Table II presents the capabilities and limitations of the second instrument which have been noted to date. In sum, the equipment is capable of performing fast, accurate analyses with a high degree of reproducibility. It can operate on either batch or continuous basis. When it is operating continuously in-line with the cleaning process, it is automatic after start-up and prints discrete analyses at less than 1 minute intervals. The mode can be selected to measure NVR in either the influent or effluent solvent or to measure the difference between the two. Operation is slower on a batch basis, requiring 20 to 30 minutes to compare a sample to a blank. The percent of error, based on analysis of prepared samples, is less than 10 percent that of the gravimetric method. Reproducibility does not vary between operators as does the gravimetric method.

TABLE II

Evaluation of Light Scattering Equipment and Technique for NVR Analysis

Advantages

1. fast
2. accurate
3. reproducible results
4. automatic operation
5. batch or continuous on-stream operation

Limitations

1. equipment mechanical maintenance
2. equipment electronic maintenance
3. matched nebulizers required for comparison mode
4. low sensitivity in 0-10 mg/500 ml range
5. low sensitivity for halocarbon NVR materials

Equipment limitations are composed principally of maintenance problems, and these are mostly electronic. Accuracy and sensitivity are good when the equipment is operating properly; however, sensitivity in the 0 to 10 mg/500 ml range, where most of the work is done, is much less than at slightly higher concentrations.

Figure 3 shows calibration curves based on prepared samples of a hydrocarbon compressor oil in trichloroethylene. Since sensitivity is a function of the slope of the calibration curves, sensitivity is about three times greater in the 10 to 20 mg range than in the 0 to 10 mg range. Also, the REL "B" curve, being steeper, shows better sensitivity than the REL "A" curve. The difference in the two curves is thought to be caused principally by differences in the two nebulizers.

The nebulizer atomizes the solvent and is a major determining factor in the properties of the aerosol. Ideally, the nebulizers should be matched so that the calibration curves are almost identical. This is especially important in the comparison mode for measuring the difference in NVR level of two solvent samples. These two curves also suggest that there is an optimum nebulizer design for maximum sensitivity of a given system.

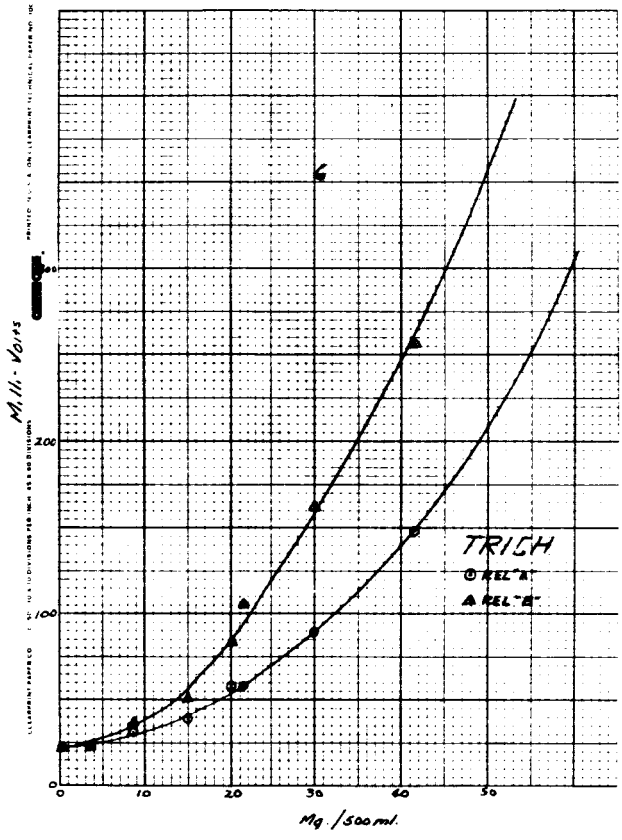



Figure 3. Hydrocarbon compressor oil in trichloroethylene

Both the technique and equipment look highly promising; however, based on the evaluation performed thus far on the prototype equipment, equipment development has not advanced to the point where it can replace the present gravimetric method. We are still evaluating the equipment and will continue working with the contractor in an effort to increase equipment reliability.

# 2. AN AUTOMATIC METHOD FOR MEASURING THE NONVOLATILE RESIDUE OF CLEANING SOLVENTS

by M. R. Jackson and M. J. Salkowski*

Presented by

M. R. JACKSON

IIT Research Institute
Chicago, Illinois

## Abstract

An instrument is described for measuring the nonvolatile residue of cleaning solvents on a continuous basis. The residue is concentrated in aerosol form by evaporation of the more volatile solvent. The volume of the concentrate aerosol is assessed by using a forward light scattering photometer. Experimentally derived curves relating photometer output to nonvolatile residue concentration are presented.

## Introduction

There is an increasing demand in the aerospace and other industries for components free as soluble residues. These range from oil-free liquid oxygen systems to flux-free radio and television circuit boards. Improved solvents and cleaning methods do much to satisfy these demands, but since economically it is usually necessary to distil an organic cleaning solvent after use so that it may be re-used in subsequent cleaning operations, there exists also a need for an instrument to measure the nonvolatile residue of cleaning solvents. Such an instrument has additional applications both in determining the relative efficiency of cleaning solvents and in following the progress of a cleaning operation.

This report describes a nonvolatile residue nephelometer developed for the NASA Marshall Space Flight Center.[1] The nephelometer will measure continuously the nonvolatile residue of cleaning solvents.

## Principle of Operation

The solvent-and-residue solution to be analyzed is aerosolized with a clean dry gas supply into a near monosized 2 to 3$\mu$ aerosol. To this is added an excess of clean solvent-free air to evaporate the solvent. The nonvolatile constituent remains as submicron aerosol in

*Now with Laboratory Equipment Corporation, St. Joseph, Michigan.

[1]Contract No. NAS8-20659.

the solvent vapor and air mixture and is passed through the sensing zone of a dark field photometer. A photomultiplier tube system detects and time-averages the total quantity of light scattered in a near forward direction by these residue droplets. The photomultiplier tube output is related by calibration to the concentration of nonvolatile residue in the solvent.

## Instrument Design

An earlier model of this instrument was described at a previous conference.[2] Modifications have been made to this design to provide easier sample handling and calibration and to make possible on-steam analysis. The instrument is shown in Figure 1.

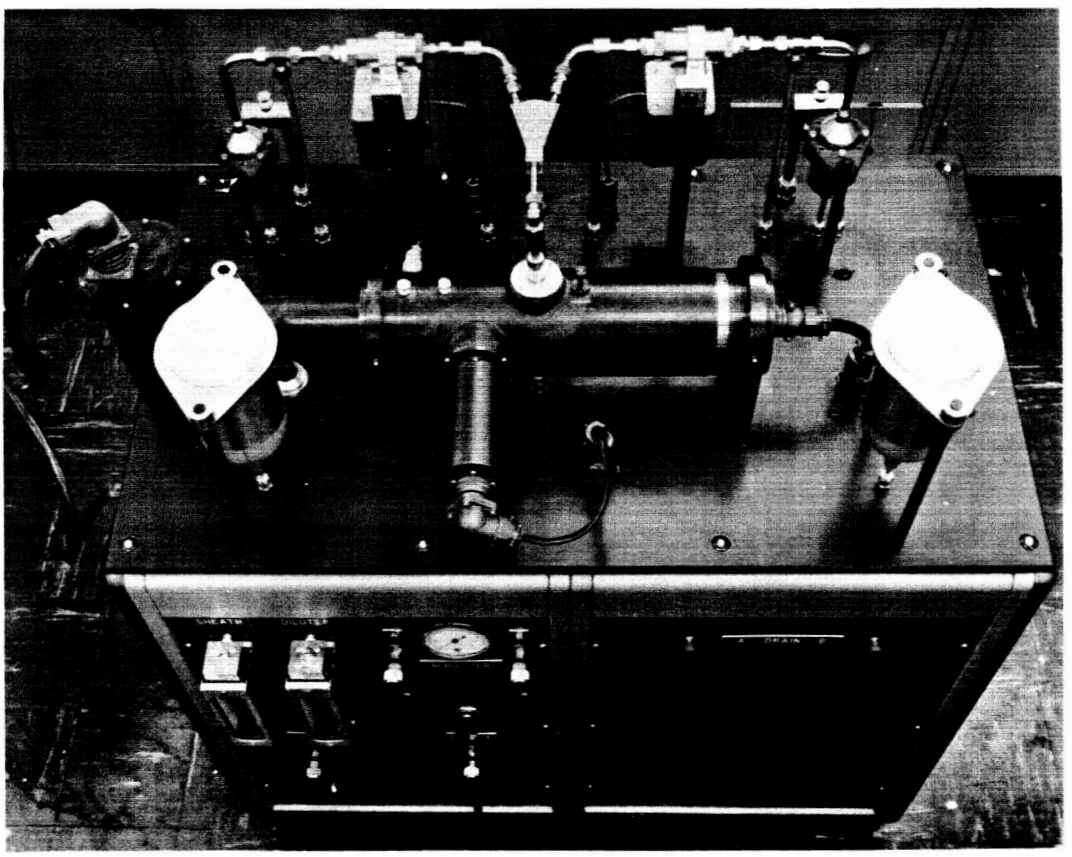


Figure 1. Nonvolatile residue nephelometer

[2] M. J. Salkowski and D. Werle, Symposium on Surface Contamination, Gatlinburg, Tennessee, June 1964, Pergamon Press.

As the schematic lay-out in Figure 2 shows, the nephelometer comprises three functional areas: (1) aerosol generation and handling, (2) photometric detection, and (3) electronic processing of data.

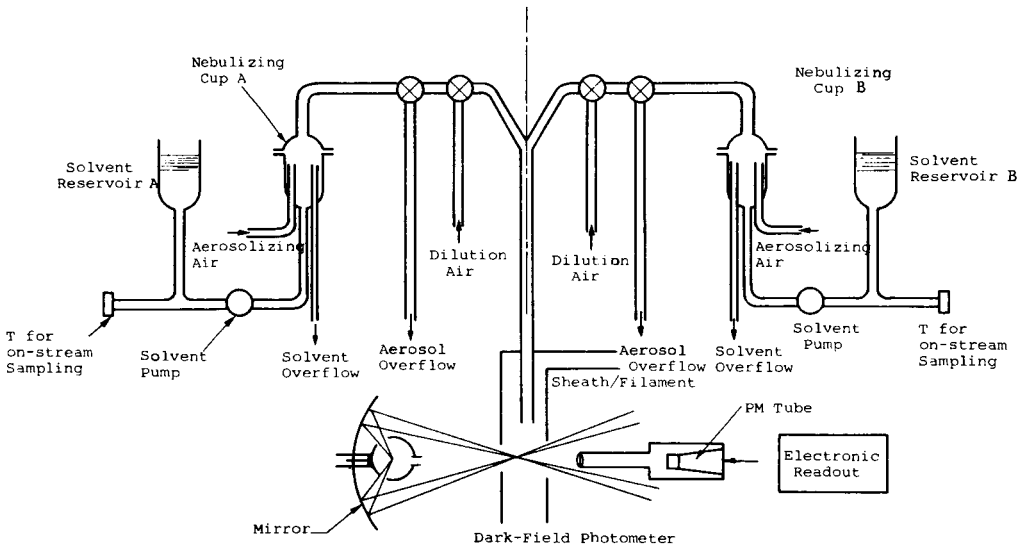



Figure 2. Schematic layout of NVR nephelometer

To generate the aerosol, the solvent is pumped from the solvent reservoir, or from a solvent stream, to the aerosolizing cups (see Figure 3) with the solvent pumps. In the cups, the solvent is aerosolized to a 2 to 3$\mu$ aerosol with a de Vilbiss nebulizer and a dry gas stream at 5 psig. To ensure that preferential solvent evaporation does not cause residue enrichment in the cups, excess solvent is fed to the cups and passed to waste. The nebulizers are operated continuously to ensure the attainment of equilibrium conditions during the sequential measurement of samples. During times when the aerosol sample is not being photometrically analyzed, the aerosol is passed to waste.

All gas supplies to the instrument and all solvent lines were filtered with membrane filters of nominal 0.45$\mu$ pore size. Filters were located after each possible source of solid contamination, i.e., pumps and valves upstream of the photometer.

After generation the aerosol is flash evaporated by the addition of a solvent-free gas to leave an aerosol comprising droplets of highly enriched solution of residue in solvent. This aerosol stream is presented to the photometer in a sheath filament configuration which allows for open transition of the aerosol tube in the viewing zone without causing droplet recirculation (see Figure 4).

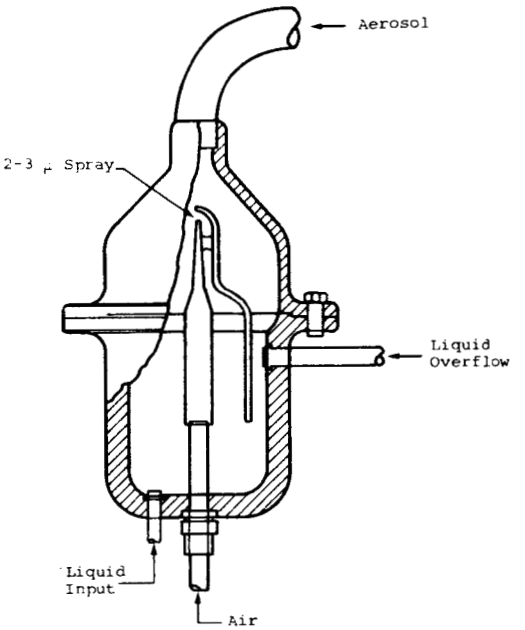



Figure 3. Aerosol generator assembly

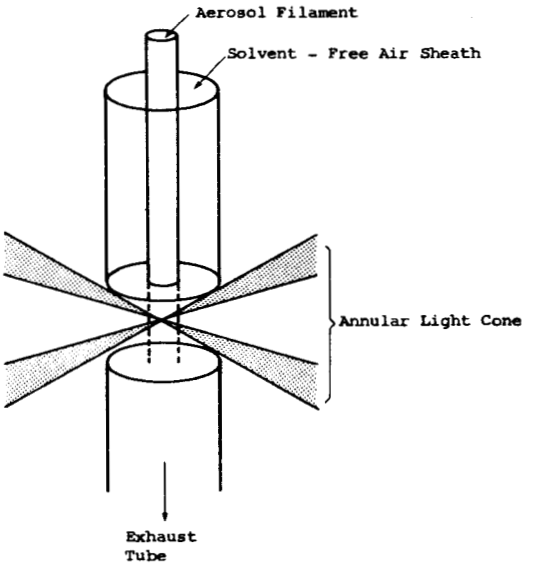



Figure 4. Aerosol filament and sheath configuration

After passing through the photometer the sheath and filament are pumped to an exhaust as a combined stream. The exhaust is run below atmospheric pressure to ensure that the aerosol sheath and filament configuration is not distributed by streaming in the event of small leaks in the photometer housing.

The cone of light, through the apex of which the aerosol passes, is produced by a coil filament lamp run off a feed-back stabilized power supply and is defined by a series of slits, as shown in Figure 2. A mirror inserted into the edge of the light cone downstream of the viewing zone reflects light into a photocell. The response of this photocell is used to control lamp intensity. The cap covering the bulb has a central hole and filter holder through which light may pass along the photometer. The center stop in the slit system contains a two position rotatable valve. In one position the axial light is allowed to pass directly to the photomultiplier tube. In the other position this light is stopped. When an aerosol is assessed, this position is selected. The other position is used during a standardization procedure.

As components age, particularly the lamp and photomultiple tube, changes occur in the amount of light falling on the photomultiplier tubes under a given condition and the sensitivity of the tube to this light. Thus it is possible for the calibration of the instrument to drift with time. The standardization procedure ensures a constant response, since both the lamp voltage, or more easily, the photomultiple dynode voltage, can be adjusted to give a constant system output when the axial light is viewed. This ensures that the total system sensitivity remains constant and, thus, the experimentally determined calibration relating system electrical output and solvent contamination concentration can be maintained even though component characteristics may change.

A chopper run at synchronous speed is located immediately in front-of the photomultiplier tube. Thus, the tube sees an AC signal. This is selectively amplified at synchronous frequency, rectified, and time averaged by using a capacitor storage system.

The photomultiplier tube output may be displayed in one of the following three ways with the system as illustrated in block form in Figure 5.

1. The photomultiplier tube output for a single nebulizer cup may be displayed directly. Either cup may be used for this function.

2. The photomultiplier tube output for both cups are displayed sequentially.

3. The photomultiplier tube output for both cups are measured and stored sequentially and their difference displayed.

In these three methods of signal presentation, the first two are used for measuring absolute solvent purity; the third method, for measuring relative purities. In this case, a known clean solvent or the solvent entering the cleaning process may be used as the standard. The cleanliness of the solvent leaving the process is compared to this standard. This mode of operation allows the progress of the cleaning operation to be monitored while the other two allow the absolute cleanliness of the component to be monitored.

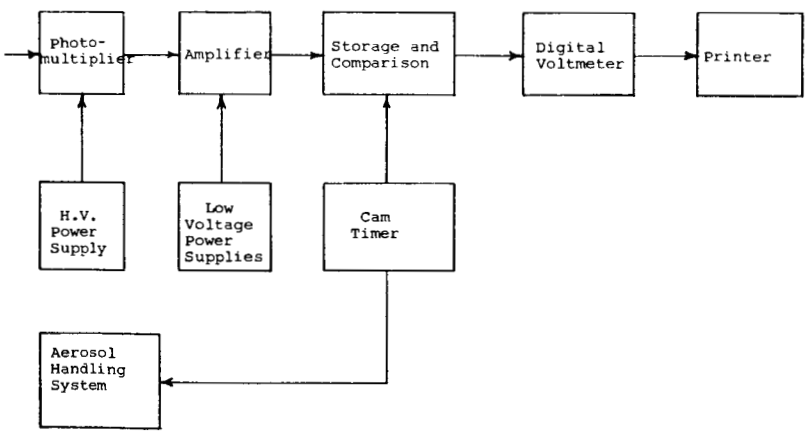



Figure 5. Electronics block diagram

## Experimental Data

The instrument was calibrated by using solutions of oil in trichloroethylene, trichloroethane, methylene chloride and freon 114. Figures 6, 7, 8 and 9 shows the experimental calibration data. A gravimetric technique was used for determing oil concentration by using temperatures near ambient.

It will be seen that differences exist between nebulizers A and B at the higher oil concentrations. These are due to dissimilarities between the nebulizer efficiencies and aerosol handling components. Two calibration curves which are reproducible for each nebulizer system are thus obtained.

These calibration curves should be used to convert photometer output to residue concentration. This is particularly important when concentration of one solvent is measured relative to a standard if the standard itself has a high contamination level. Should any components in one unit be changed, the calibration curve may also change, but recalibration can readily be carried out by using the known calibration of the other nebulizer system. Thus, this provides a continuous or batch measurement of nonvolatile residue contaminants for application to low temperature boiling point solvents.

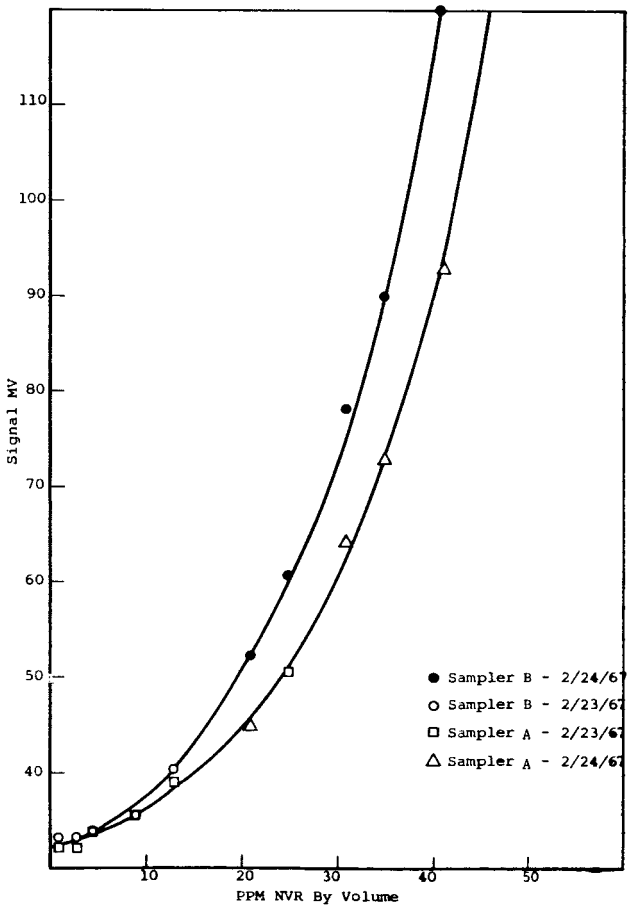



Figure 6. Trichloroethylene calibration

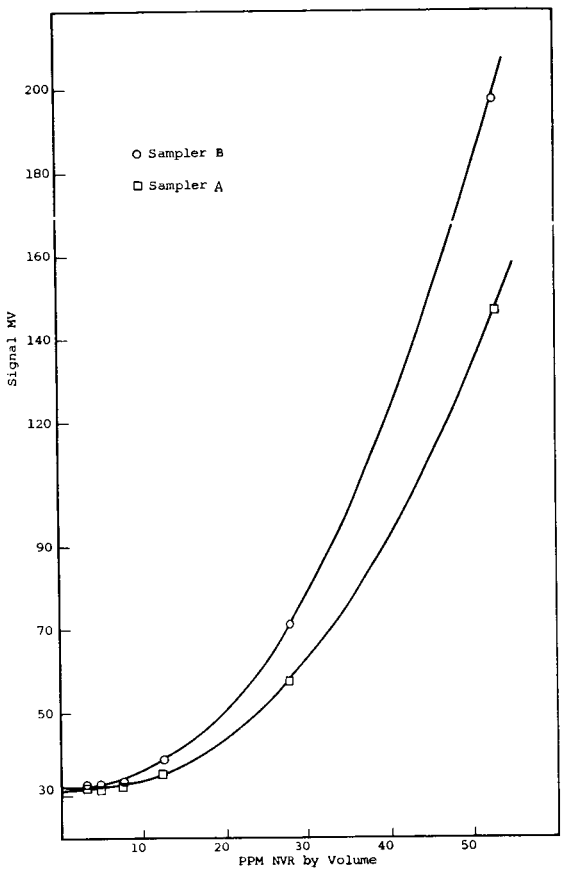



Figure 7. Trichloroethane calibration

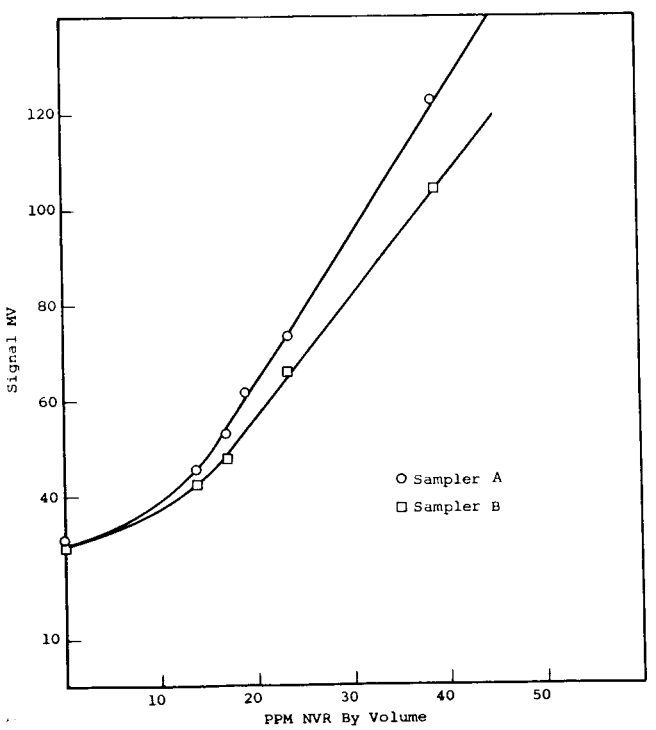



Figure 8. Methylene chloride

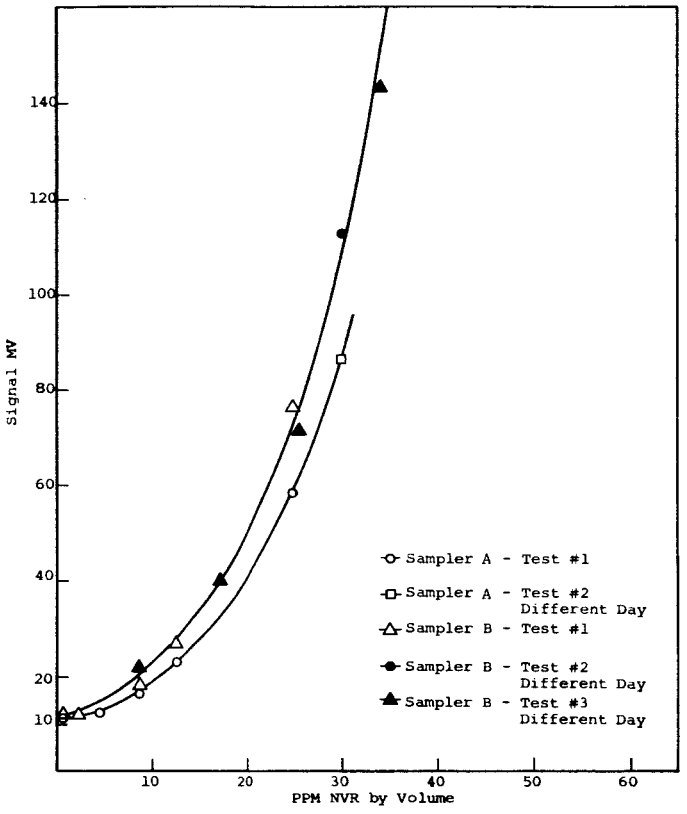



Figure 9. Freon calibration

# 3. THE SOLVENT PURITY METER AND ITS APPLICATIONS TO PRECISION CLEANING

by

F. W. OSWALT

Manufacturing Research Division
Sandia Laboratory
Albuquerque, New Mexico

Abstract

Solvent Purity Meters have been used at Sandia Corporation since May 1965 primarily to monitor the nonvolatile residue content of cleaning solvents. Moreover, they have been used and found valuable in seven separate applications by twelve different groups within the past year. Six different solvents have been monitored in conjunction with the cleaning of assorted piece parts; hydrocarbons have been the most common contaminants encountered.

Results of uses to date show the instrument reliably measures the soluble residue content of volatile cleaning solvents and gives a useful indirect index of cleanliness to the product being processed. It gives an instantaneous and repeatable readout of residue as low as one part per million and has been used either for batch sampling or as an on-line continuous monitor for recirculating cleaning systems.

No major malfunctions or operator problems have been noted. Definite cost savings are possible through the use of the meter. The units used to date have been development models; however, commercial versions are now available.

## Introduction

In March 1965 Sandia Corporation began work on a Solvent Purity Meter (SPM) for monitoring the nonvolatile residue content of cleaning solvents. The design was based on the principle of aerosolizing a solvent and, by using light-scattering techniques, measuring the nonvolatile residue (NVR) content with a photometer. This principle had been described previously at the 1964 International Symposium on Surface Contamination[1] and Sandia's development of a prototype tester was covered in a presentation at the 1966 Technical Meeting of the American

[1] Salkowski and Werle, Nonvolatile Residue Nephelometer. Presented at the International Symposium on Surface Contamination, Gatlinburg, Tennessee, June 8, 1964.

Association for Contamination Control.[2] A report summarizing one year's experience on the utilization of the development instruments at various suppliers was presented at the 1967 Technical Meeting of the American Association for Contamination Control.[3]

The objectives were to maintain a low sensitivity level of about one part per million (ppm), to provide instantaneous and repeatable readout, and to do this in a simple-to-operate manner so that production personnel could use the instrument.

## Description of Equipment

In May 1965 a development model (see Figure 1) was put into operation at a supplier facility. The photometer[4] is in the upper portion of the figure, and the aerosolizing and solvent-handling apparatus in the lower part. The photometer requires 115 volts AC at 5 amperes, and the aerosolizing equipment requires approximately 2 cubic feet per minute (cfm) of building air or dry nitrogen. An in-line absolute filter cleans the air or nitrogen before use.

As shown schematically in Figure 2, the solvent is introduced into the nebulizer (atomizer) by use of a 50 cc syringe and a two-way check valve. The solvent is then atomized and passed into the drier, where 2 cfm of clean air or nitrogen is added. The drier output is sampled by the forward-light-scattering photometer. The logarithmic characteristic of the photometer readout is ideally suited to solvent purity testing, since the readings of greatest interest are at the low end of the scale.

Any of three methods may be used to supply solvent to the system. The solvent may be poured into the syringe from a beaker or similar small container, it may be hand-pumped from a drum or tank by use of the syringe, or it may be continually forced through the system. Calibration is accomplished by adding known amounts of a soluble contaminant, such as hydrocarbons or solder flux, to a reagent-grade solvent and noting the photometer readings in ppm for the given amounts of contaminant. A typical calibration curve is shown in Figure 3; the abscissa denotes the photometer reading, and the ordinate represents the NVR content in ppm. Concentrations greater than 100 ppm are diluted with clean solvent so that on-curve readings may be obtained.

---

[2] R. C. Marsh, Cleanliness Meter and Its Application to Solvent Cleaning. Presented at the 1966 Technical Meeting of the American Association for Contamination Control at Houston, Texas.

[3] F. W. Oswalt, One Year's Experience with the Solvent Purity Meter. Presented at the 1967 Technical Meeting of the American Association for Contamination Control at Washington, D. C.

[4] Aerosol Dust and Smoke Photometer, Model JM2000, Phoenix Precision Instrument Company, Philadelphia, Pennsylvania.

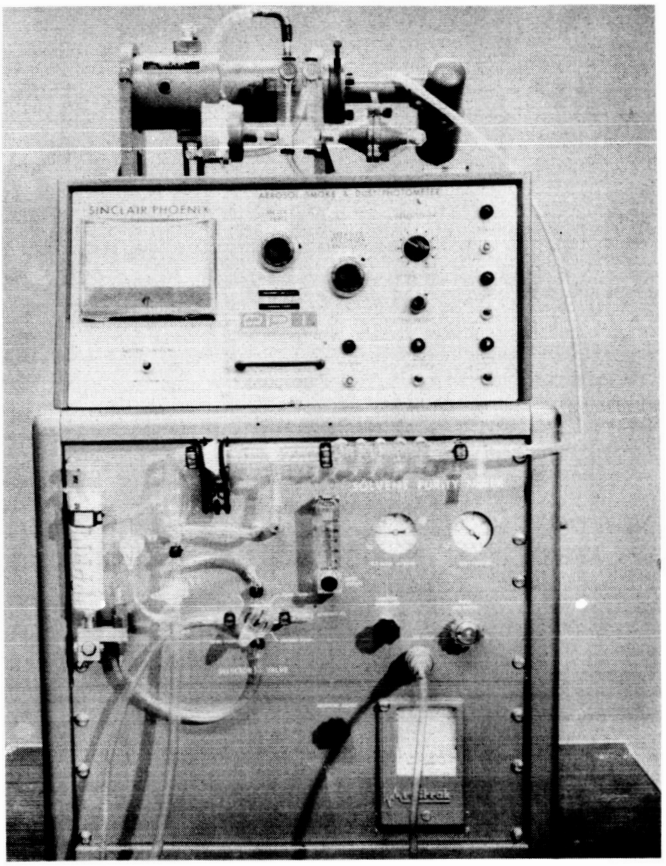


Figure 1. Development model of solvent purity meter

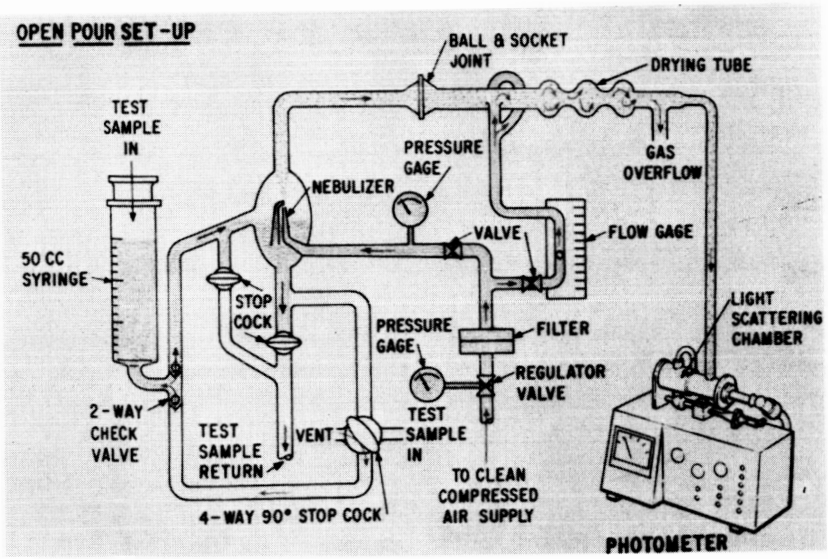



Figure 2. Flow schematic of SPM

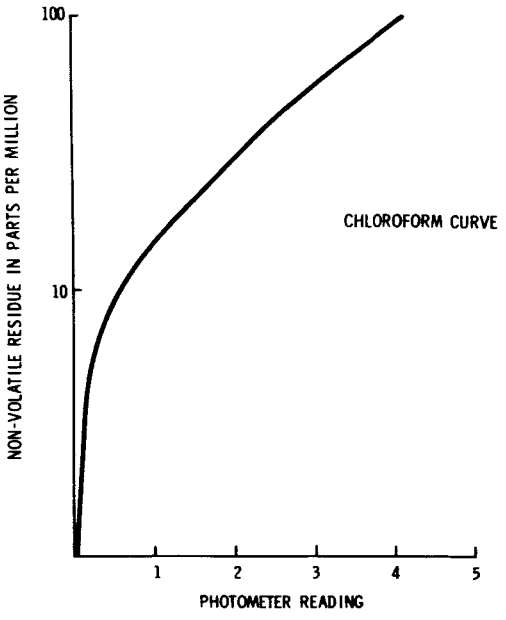



Figure 3.

Typical calibration curve

The SPM has been used at Sandia Corporation to monitor trichloroethylene, trichlorotrifluoroethane (TF), isopropyl alcohol, deionized water, chloroform, and trichlorotrifluoroethane-methylene chloride. Parts cleaned include small watch parts, machined components, electromechanical subassemblies, high-pressure system parts, vacuum system parts, printed circuit boards, optical assemblies, and aerospace components.

Applications of the SPM in cleaning processes are as follows.

## Verification of Purity of New Solvent

Verification of the purity of new solvents (as received) has often been neglected because of the processing time involved. At Sandia, the SPM is being used to monitor the purity of the solvent by taking a sample directly from the shipping container. The solvent is hand-pumped with the syringe, with only the sample hose in the container, thus eliminating possible sources of contamination such as beakers and pouring. Thus far, there has been no reported instance of high contamination in a new solvent; however, this does not minimize the need for monitoring. The one minute required to make the test is considered well spent.

## Verification of Purity of Redistilled Solvent

Closely related to the above application is use of the SPM to monitor the distillate in redistillation processes, both those incorporated into ultrasonic cleaning systems and those that are high-volume stills. Not only does continuous monitoring verify purity at any given time, but the SPM is a very useful tool for evaluating the capability of the distillation process when extreme loads of contaminants are present in the boiling sump.

## Determination of Part Cleanliness

Part cleanliness determination is another area in which much work has been done, and yet no generally acceptable method is existent. The SPM is not the answer to this problem; however, it does have an application in this area and is so used. Monitoring is done at the final cleaning stage of small parts that are beaker cleaned. The purity of the solvent in the beaker is monitored before cleaning; after cleaning, the solvent is again monitored. If there is an increase in contamination in the solvent, it is known that the part cannot be clean, since there will be dissolved contaminants in the solvent adhering to the part after it is removed from the beaker, and these contaminants will remain on the part after the solvent dries. In cases where there is no contaminant increase after cleaning, one can only assume either that the part is clean or that there is a contaminant on the part that this cleaning stage will not remove.

## Development of Cleaning Processes

The SPM is being used to develop cleaning processes on the same principle as discussed in the preceding paragraph. A number of like parts are cleaned until no contaminant is being removed. These parts are then dipped in a diluted mixture of solvent and the most likely contaminant, and are then baked. They can now be used to determine the relative effectiveness--in terms of equipment, time, and solvents required--of various cleaning processes.

## Educational Tool

Perhaps one of the most under-rated applications of the SPM is as an educational tool for production workers. People become much more aware of the need for and the validity of recommended contamination control practices when the effects of using an unclean beaker, placing a finger in the solvent, and similar practices are illustrated.

## Troubleshooting of Cleaning Processes

Another application is troubleshooting of cleaning processes. In a typical example, a plant used a central solvent supply to pump solvents to all cleaning stations from this single source; the SPM was being used to verify solvent purity at the cleaning stations. Suddenly, a significant decrease in solvent purity was noted at all cleaning stations. A check of the solvent in the central storage tanks showed that the NVR at this point was well within allowable limits. Downstream from the pump in the system was a filter to remove particulate contamination. It was found that the replacement of this filter had just been completed at the time solvent contamination was first noted. Further tests and subsequent contact with the filter manufacturer indicated that a contaminant, possible uncured epoxy, was being dissolved from the new filter by the solvent.

## Determination of Acceptable Rate of Cleaning

The use of cleaning systems with built-in redistillation has raised the question of what cleaning rate can be maintained without a build-up of NVR in the cleaning tank. The SPM has been used in establishing the quantities of parts that may be cleaned in a given time.

## Maintenance

Maintenance of the instruments thus far has been limited to replacement of broken glassware on two occasions, repair of minor leaks in air-handling lines in one case, and adjustment of the photometer. It appears that maintenance is not a major problem.

## Evaluation

It is well to emphasize that the SPM is sensitive only to soluable contaminants and is not capable of counting particulate matter suspended in solvents. A cross-check was made with three different meters and three different operators to assay the effects of similar items of equipment and the human factor aspects. Four of the more common solvents were used in each meter. Calibration curves were prepared for each solvent used in each instrument. Figure 4 shows the three curves of one of the solvents plotted on a single graph; as can be seen, there is some difference in the curves. However, when an unknown amount of contaminant was added to a reagent-grade solvent and this solvent was tested in the three instruments, the NVR readouts in ppm were found to be nearly identical. Therefore, it is thought that no significant equipment or operator error exists.

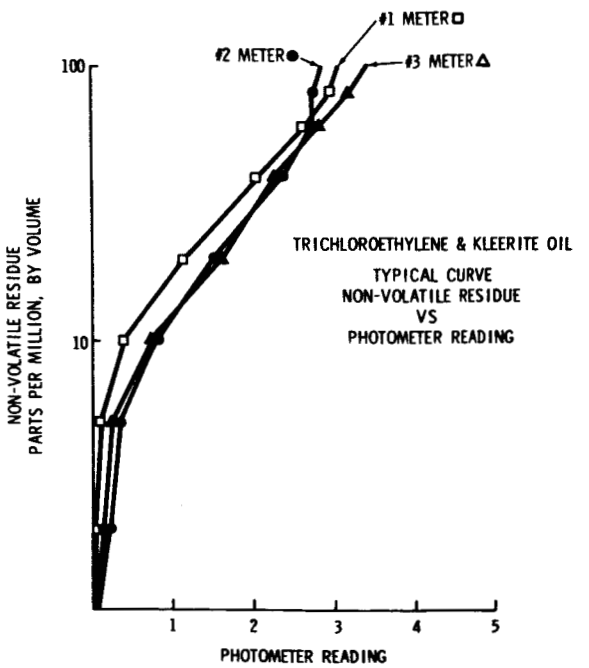



Figure 4.
Comparison curves for 3 SPM's

The results obtained from this method of determining the NVR in solvents have been compared to results obtained with procedures in ASTM Standard No. D1353-64. The correlation between results shows that the SPM method is accurate within the limits of laboratory requirements.

There has been a question as to variation in the curves when different contaminants are present in a solvent. Tests using TF as the solvent, with light-weight lubricating oil, solder flux, and a combination of the two as the contaminant, resulted in curves that varied slightly from one another. These variations are no cause for concern; however, it is suggested that the contaminant most likely to be encountered in practice be used in calibration.

## Looking Forward

Because of the intense interest in the development models exhibited by Sandia suppliers, NASA groups, AEC integrated contractors, and the aerospace industry, Sandia recognized that the next step should be to establish commercial suppliers for the instrument.

At the present time, SPM's are available from two commercial suppliers. A third firm has exhibited interest in marketing the instrument, but to date has not done so. Figure 5 is a picture of a commercially available instrument using equipment nearly identical to that in the development models.

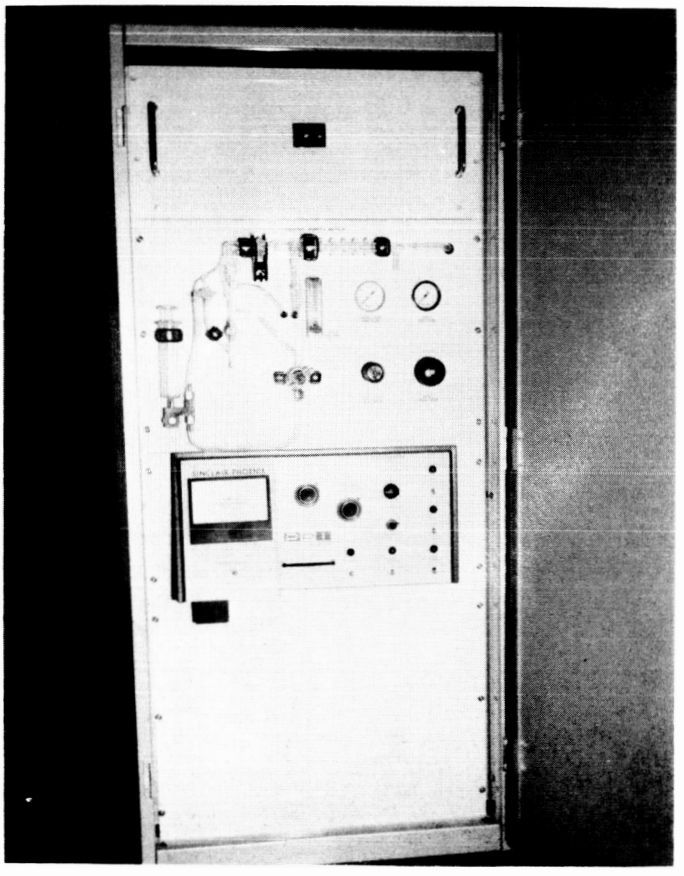


Figure 5. Commercial version of SPM

It is easily noted that instruments manufactured by a company with packaging knowledge and experience have a much more professional appearance. Figure 6 is a picture of another commercially available instrument; in this case, the manufacturer designed and fabricated his own forward-light-scattering photometer. In both instruments, the capability of using the photometer to leak test High Efficiency Particulate Aerosol (HEPA) filters is retained.

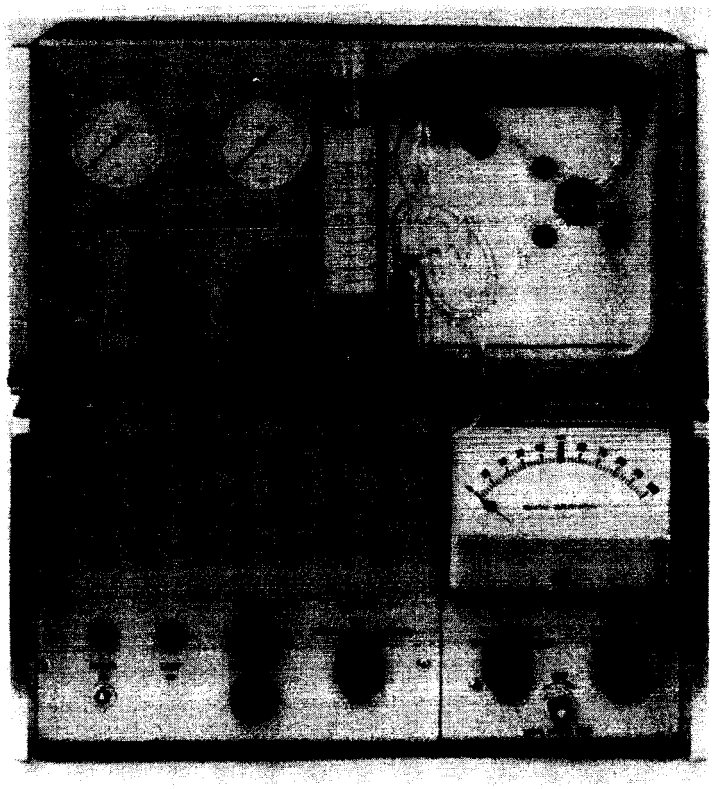


Figure 6.
Commercial version of SPM

There has been some discussion of a combination meter that can be used to determine NVR and also to count liquid-borne-particles; since cleaning processes are becoming more exact, a device such as this may appear in the future. Although the solvent suppliers have not as yet used the instrument as a production monitor, it is believed that this application is imminent. Some thought has been given to increasing the sensitivity by improving the photometer; however, no demand for greater sensitivity has arisen, and thus no efforts have been expended in this direction.

## Conclusions

The Sandia experience with the SPM has shown it to be a valuable tool for use at facilities performing precision cleaning. Solvents

currently being monitored with the SPM are (1) trichlorotrifluoroethane, (2) trichloroethylene, (3) isopropyl alcohol, (4) deionized water, (5) chloroform and (6) trichlorotrifluoroethane-methylene chloride. The applications investigated with the SPM are (1) verification of new solvent purity, (2) verification of redestilled solvent purity, (3) part cleanliness determination, (4) develop cleaning process, (5) educational tool and (6) troubleshooting cleaning processes.

The instrument has proven to be a valid, reliable, and repeatable device for measuring instantaneously the nonvolatile residue content in volatile cleaning solvents. This measurement has been a useful indirect index of the cleanliness of the product being processed.

# 4. CONTAMINATION SENSORS

by

H. V. Anthony, J. G. Drew, E. F. LaRue,
J. P. McDonald, and M. Piccone

Presented by

M. PICCONE

Martin Marietta Corporation
Denver, Colorado

Many components used in rocket propulsion systems and spacecraft are subject to malfunction or subnormal performance if excessive contamination exists within the system, or if the purity of the fluid is below a certain value. Because of the high reliability required in the functioning of these components, contamination control plays a very important part in the successful performance of rocket propelled boosters and spacecraft.

This paper presents the results of a NASA-sponsored study, completed in April 1966, intended to provide a basis for possible eventual use of automatic contamination sensors and monitors capable of being installed in a fluid system, and of remotely indicating particle-contamination count, amount of moisture present, purity of the fluid, or some combination of these. Some information concerning mechanical sampling is also included.

The fluids discussed in this paper are liquid oxygen, liquid hydrogen, liquid nitrogen, helium, gaseous oxygen, gaseous hydrogen, gaseous nitrogen, RP-1, 50/50 hydrazine/unsymmetrical dimethylhydrazine, monomethyl hydrazine, and nitrogen tetroxide.

A certain ambiguity is associated with the terms sampling and monitoring. As used in this paper sampling is defined as withdrawing a volume of fluid, or of some constituent of the fluid, including particles, from the main fluid stream, and transporting it to another location for analysis. Monitoring is defined as attaching to the main fluid stream a device capable of measuring and reporting the variation of a significant parameter of the fluid as a function of time.

The terms purity, impurity, assay, and particulate contamination also require definition. Purity refers to the concentration of the desirable constituent in the fluid, e.g., 99.6% oxygen. Impurity refers to the concentration of undesirable constituents in the fluid, e.g., 0.1% nitrogen in liquid oxygen. Assay refers to the proportion of desirable constituents in a mixture, e.g., 50% hydrazine - 50% UDMH. Particulate contamination refers to particles in the fluid that are solid at normal room temperature.

During the study program a survey was made of the launch, static test, and aerospace company facilities in the United States where the fluids of interest to the study were utilized. The information obtained included sampling procedures, sampling equipment, fluid criteria, and automatic monitoring equipment. Manufacturers of instrumentation and sampling equipment were also contacted to obtain information concerning the instrumentation and sampling equipment readily available, possible modifications to existing equipment, and conceptual ideas which might form the basis for development of instrumentation to measure parameters not capable of measurement with presently available equipment.

## Mechanical Sampling Devices for Purity Parameters

Mechanical sampling for purity parameters (as distinguished from particulate sampling) cannot practicably be improved upon for the majority of the fluids. Existing devices, such as the Cosmodyne Cryogenic Sampler for the cryogenic liquids, the double valved stainless steel pressure bomb for the gases and the storable propellants (nitrogen tetroxide and the hydrazine-type fuels), and a simple glass bottle with screw-on bakelite cap and conical polyethylene seal for RP-1, are quite satisfactory. Although some minor improvements are possible, no significant changes to existing equipment are recommended.

## Mechanical Sampling for Particulate Contamination

In general, sampling for particulate contamination is accomplished by one of two general procedures. The first is to take a fluid sample in a container, filter this sample through a membrane that has a counting grid imprinted on it, and rinse the membrane and transfer it to a microscope stage for counting. The second is to take a particle sample from the fluid directly on the counting surface, which is evaluation-rinsed or precounted before assembly, transported to the sampling site, connected to the sampling point, disconnected from the sampling point, and transported back to the laboratory (in most instances in uncontrolled orientation with respect to the vertical). In the laboratory, the sampling device is disassembled, and the membrane is transferred to the microscope stage for counting. The most significant problem that accompanies these operations is the fact that particle generation can occur in so many ways. Particles are generated or transferred each time two surfaces touch, by mechanical and chemical attrition of the surfaces (abrasion, corrosion), by electrostatic attraction, and by cohesion or adhesion. Particles are borne by air currents and by Brownian movement, and transferred by condensation. In fact, many naturally occurring physical and chemical changes are attended by particle generation or transfer.

Thus, the most difficult part of particle sampling is not the sensitivity of the detection and sizing method, but rather the exclusion from the counting surface of particles that were never in the operational fluid stream. These occur on the counting surface before sampling and on the flow-contacting surfaces of the sampling-point connections and of the sampling tool. They are accidentally transferred to the counting surface during installation of the counting surface into the sampling tool, during its removal from the tool and during counting. They are generated by abrasion of mechanical components

during installation and removal of the counting surface and the sampling tool. They are transferred from the flow-contacting surfaces of sampling and sample-transfer equipment in those cases where the fluid is returned to the laboratory for filtration and counting. They are transferred from adjacent surfaces, from the atmosphere, and from personnel during all times that the sampled fluid or the counting surface is exposed.

These contributions do not seriously affect total-filterable-solids determinations, but they frequently produce a serious effect on particle size and count determinations.

In view of this, exposure of the sampled particulate matter to environmental contributions should be minimized by filtering for particle-count criteria at the sampling site; by precounting the membrane after the sampling tool is connected to the sampling point and an initial operational fluid flow has occurred through it; by sampling the flow and counting particles without disturbing or transporting the sampling device between these operations; and by taking other allied precautions that would minimize the contribution to the sample from sources other than the operational fluid. The device proposed to accomplish this is the in-line filter holder and counter (ILFHC).

Figures 1 through 5 show a high-pressure configuration of the ILFHC and accessories designed for maximum portability, ruggedness, and effective containment of high pressure, toxic, reactive or cryogenic operational fluids.

Figures 1 and 2 show the filter holding and counting chamber, including the arrangement of the metallic membrane and support screen, the belleville spring seal, the spring loaded ball bearing, optical window and inlet tube, strongback supports, and circumferential clamp.

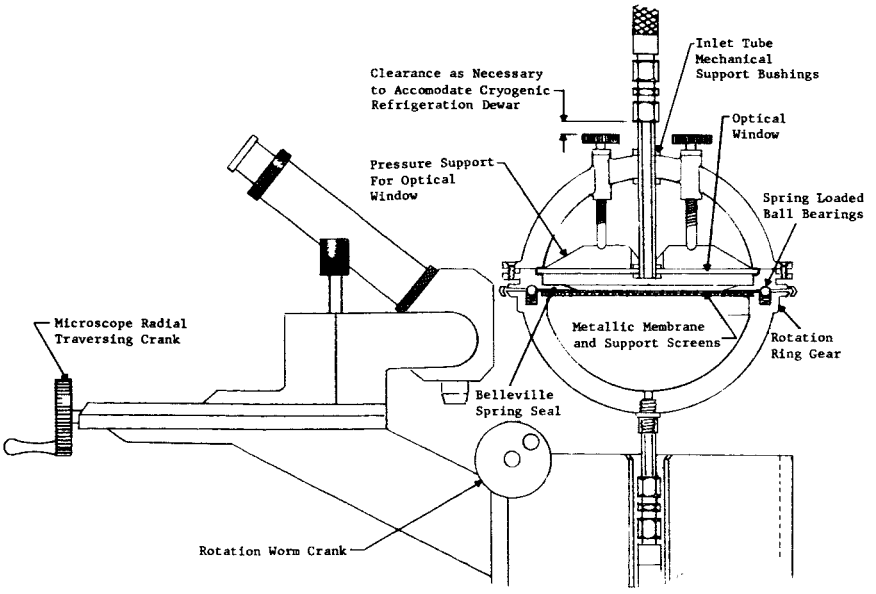



Figure 1. High-pressure ILFHC positioned above microscope fixture

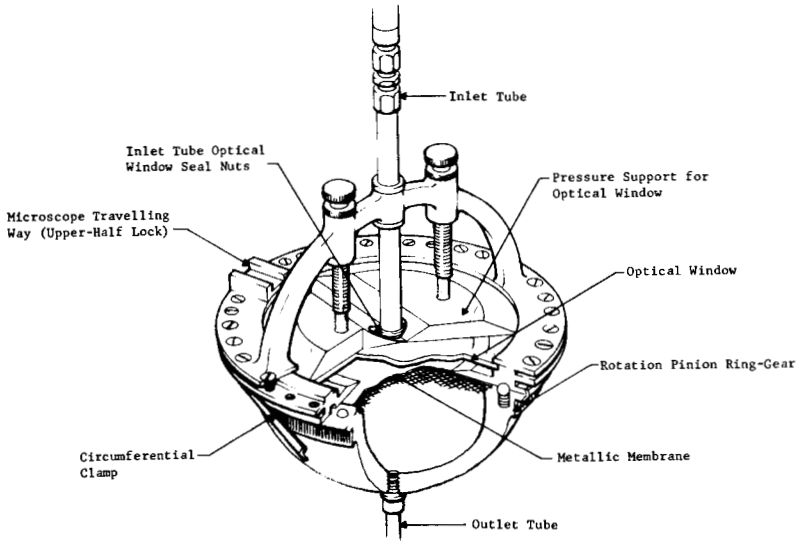



Figure 2. High-pressure ILFHC chamber

For ambient liquids or gases, the circumferential clamp is tightened prior to purge-flow and prior to sample-flow. This forces the knife-edge of the belleville spring to seal against the nonporous circumferential zone of the metallic membrane. The strongback supports are placed against the optical window and force is applied to them by means of the strongback clamps until they bottom out, metal to metal, on the inlet tube optical window upper seal nut. The face of these supports applied to the window is a resilient plastic.

Sample flow is distributed radially through the porous annular zone of the metallic membrane from the concentrically positioned inlet-tube outlet.

When flow (and purging as required) is completed, the strongback supports are removed and the circumferential clamp is loosened. This allows the spring-loaded ball bearings to lift the upper chamber half until the knife-edge seal of the belleville spring just touches the membrane, with minimum friction, so that the lower chamber half can rotate with respect to the upper.

This rotation is effected by the rotation worm crank, shown as a portion of the microscope fixture, which engages the ring gear (an inset pinion-stock gear) just outboard of the spring-loaded ball bearing sockets. The microscope radial traversing worm gear moves the microscope and its focusing mechanism radially with respect to the membrane and chamber so that controlled relative movement between the microscopic field and filtration surface is established in polar coordinates. Full field or "statistical" counting of the filtration surface may be performed.

Note, in Figure 2, the upper chamber-half rotational lock which engages the upper microscope traveling way when the microscope is projected radially onto the chamber. This serves to prevent rotational movement of the upper chamber-half with respect to the microscope when the lower chamber-half is rotated.

Figure 3 shows the holding cylinder for the filtration/counting chamber, the attached microscope, microscope mount, traversing and focusing assembly, the rotational worm crank and a phantom view of the rotation worm assembly. The lower half circumferential seal flange of the filtration/counting chamber bears on the support shoulder forming the planar top of the holding cylinder. By this arrangement the rotating ring gear of the former engages with the rotating worm gear of the latter.

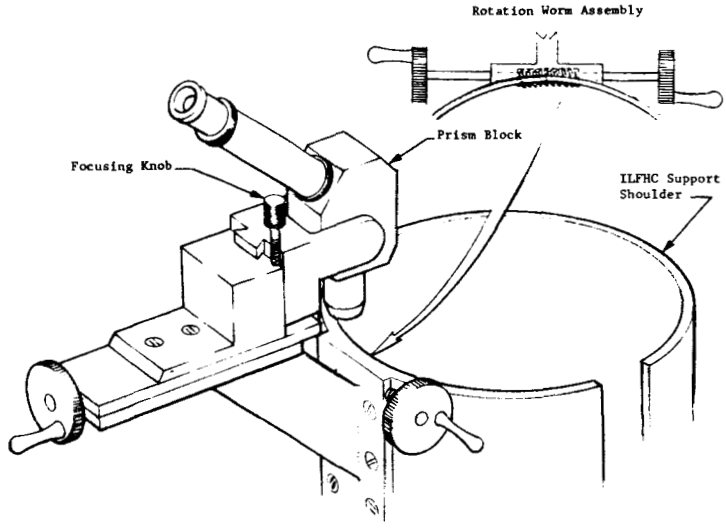



Figure 3. Pictorial view of microscope fixture for high-pressure ILFHC

Figure 4 shows a double-walled insulating jacket used to effect cool down of the ILFHC preparatory to filtration of $LO_2$, $LN_2$, and $LH_2$.

This device, hinged on one side, is closed around the ILFHC chamber and seals to it by semi-cylindrical elastomeric inserts in the dewar halves which compress against the inlet and outlet tubes of the filtration/counting chamber. Cryogen is then allowed to flow through the annular space between the chamber and the dewar until the chamber temperature is at the boiling point of the cryogen, after which sample flow is commenced through the filtration/counting chamber. Figure 5 shows the ILFHC as it might look while in use.

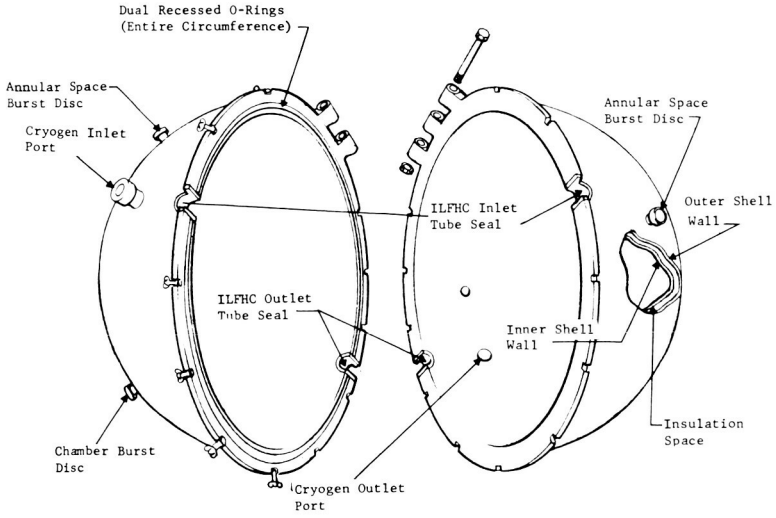



Figure 4. ILFHC cryogenic refrigeration dewar

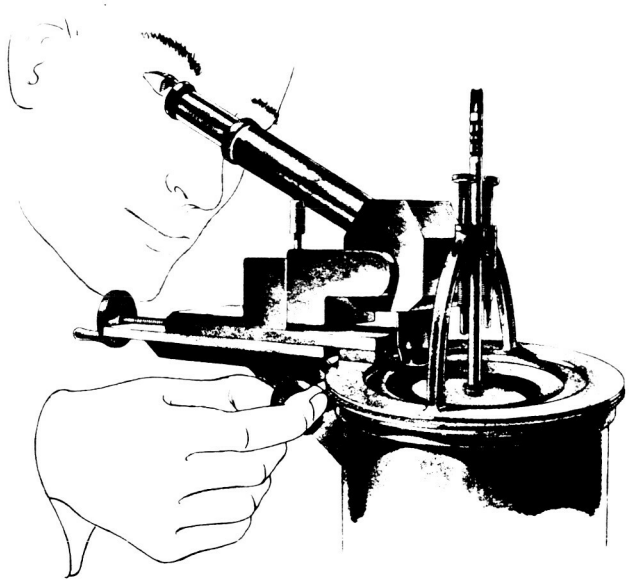


Figure 5. ILFHC in use with attached microscope

## Particle Monitoring

At present, there are four operable concepts of automatic particle counters. The HIAC Automatic Particle Counter, Figure 6, detects the projected cross-sectional area of particles passing between the light source and the phototube by measuring the momentary reduction in the output signal from the phototube caused by the particle shadow. The manufacturer believes that this instrument could be adapted for cryogenic usage and for the gases included in this study. RP-1 and $GN_2$ have been monitored by this instrument, but its adaptability for $N_2O_4$ and the amine fuels is not known.

Cells are available which count particles in selected size categories from 85 to 2500 $\mu$ with a capability of up to 12 particles/cc without coincidence, and from 35 to 1000 $\mu$ with a capability of up to 75 particles/cc without coincidence, at a flow rate of 3 liters/minute. All of the fluid presented to the instrument is sensed and none is bypassed.

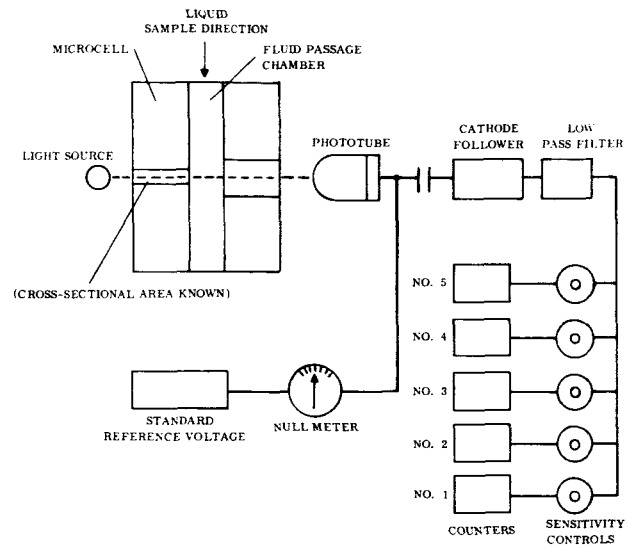



Figure 6. Schematic diagram of HIAC automatic particle counter

The Royco Particle Counter, Figure 7, detects the light scattering by the particles at 90 degrees to the incident light. The intensity of the scattered light is sensed by the photomultiplier tube whose output is amplified and discriminated and sent to the size range counters. The manufacturer believes that the instrument can be adapted for cryogenic usage, that it can be used for the gases included in this study, and that it appears feasible to use it for particles larger than 10 $\mu$ in $N_2O_4$. It is used for RP-1 and for gases up to 25,000 psi. The amine fuels have not been investigated. The flow rate of the liquid cells is 100 cc/minute and can be raised to 1 gal/minute with some loss of

sensitivity. (The sensitivity is for particle categories considerably smaller than those in the fluids in this study.) The flow rate of the gas cell is 1/10 standard cu ft/minute. There is no internal bypass in the instrument.

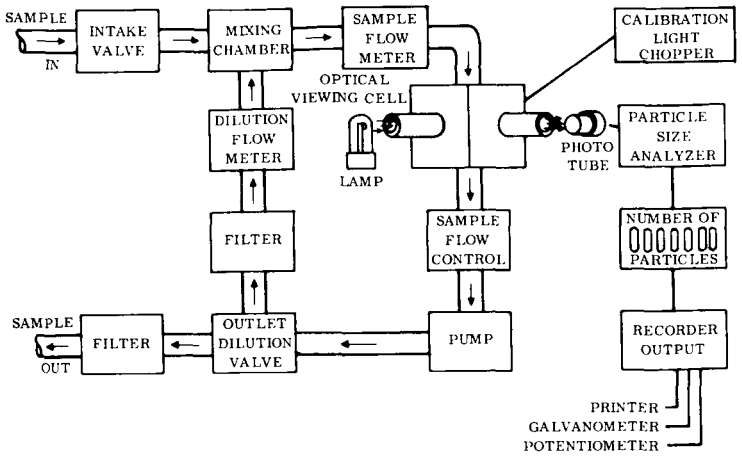



Figure 7. Schematic diagram of the optical sensor portion of the Royco nephelometer liquid borne particle monitor

The MicroScan Continuous Contamination Monitor, Figure 8, measures the capacitance change of the sample stream due to particles and free water. The sensors consist of two capacitor plates between which the sample stream passes. The readout is a function of the total volume of the contaminant and thus no size or count is sensed. Particles larger than 200$\mu$ are separated before reaching the sensing chamber. A portion of the sample stream bypasses the sensing chamber.

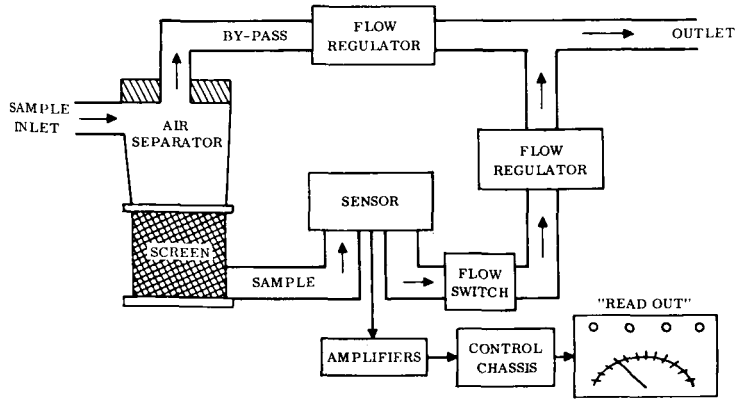



Figure 8. Schematic diagram of the MicroScan

The manufacturer states that the instrument has application for RP-1 and possibly the amine fuels, but applicability to any of the remainder of the fluids is doubtful.

The Sperry Liquid Contaminant Level Indicator, Figure 9, senses particles in a fraction of the cross-sectional area of the sample stream by measuring the amplitude of a reflected 5.0 mc ultrasonic pulse produced by a lithium sulphate transducer.

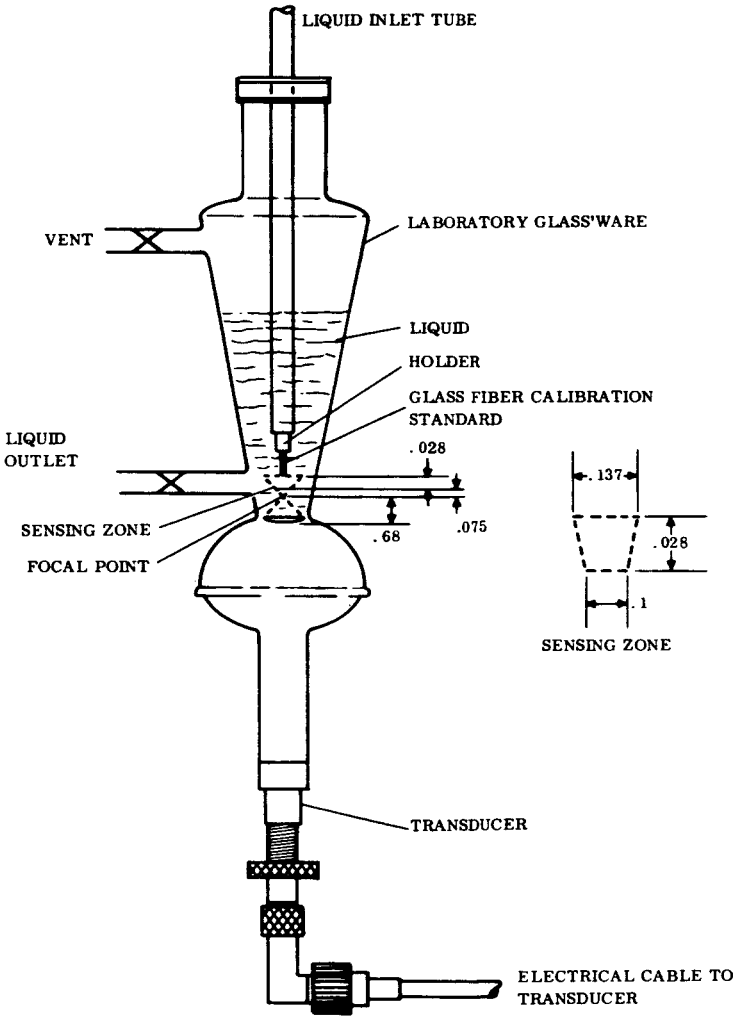



Figure 9.
Sensing zone of the Sperry Liquid Contaminant Level Indicator

None of the automatic particle counters described can detect or report the longest dimension of the particle or fiber. Since most, if not all, existing contamination control criteria are defined in terms of the longest dimension, new criteria would have to be developed to permit use of the automatic counter, or a correlation would have to be demonstrated between particle size and count, determined by the automatic counter, and size and count determined by the light microscope method (SAE ARP 598).

It thus appears that modifications to existing designs may be necessary to permit automatic monitoring of particulate contamination in the fluids discussed in this paper. Nevertheless, the advantage to be gained from the use of automatic particle counters in fluid systems, so integrated into the system that flow will be automatically stopped if excessive contamination is detected, is obvious. It is astonishing that the adaptation of automatic particle counters to fluid systems associated with large rocket test facilities has been so long neglected.

## Isokinetic Sampling

The foregoing discussions on particle sampling and monitoring assume that the particulate matter in the fluid being sampled or monitored is representative of the main fluid stream. Workers at Oklahoma State University have shown that, for particles smaller than 100$\mu$ in fluids as viscous as MIL-H-5606 hydraulic oil, simple side tap sampling of turbulent streams will give representative samples of particle contamination (Reference 1). However, Wyle Laboratories has shown that, for water and gases, isokinetic sampling devices are required if representative samples of particle contamination are to be obtained (References 2 and 3). Our recommendation, therefore, is that all samples taken from cryogenic fluid streams and all particulate samples taken from the remainder of the fluids while in operational flow be taken through a Wyle Laboratories dynamic fluid sampler or a Maledco Engineering Company turbulent flow sampling valve, depending on the line sizes and fluids involved.

The Wyle unit incorporates two basic principles: (1) inclusion of a mechanism which permits the withdrawal of a "slice" of fluid from the full cross section of the fluid system; (2) maintenance of isokinetic flow conditions both inside and outside of the sample withdrawal mechanism. The Maledco unit is designed with a "Z" section flow path which creates turbulence, and the sample is withdrawn directly from the turbulent section.

Figure 10 shows a partially exploded view of a Wyle dynamic fluid sampler. The inlet face of the wedge, which is the second item in the center lower foreground, can be clearly seen. Figure 11 shows a sketch of the dynamic fluid sampler installed in a line for automatic inline sampling of the fluid stream. The differential pressure transducer senses the difference between the static line pressure and the total pressure within the sample withdrawal mechanism. Therefore, when the differential pressure is zero, the mainstream velocity and the sample velocity within the sampler withdrawal mechanism are equal and isokinetic flow is attained.

---

[1] R. E. Reed, R. H. Tucker, and K. Stone: Study of Filtration Mechanics and Sampling Techniques. Oklahoma State University, Stillwater, Oklahoma, Technical Summary Report, 30 November 1964.

[2] L. N. Mortenson: Evaluation Testing of a Flow Sampling System Designed by the Fluor Corporation Ltd. AFBMD-TR-61-38. Wyle Laboratories, El Segundo, California, 20 March 1961.

[3] L. N. Mortenson: Evaluation Testing of a Flow Sampling Wedge. AFBSD-TR-61-53. Ibid., 17 July 1961.

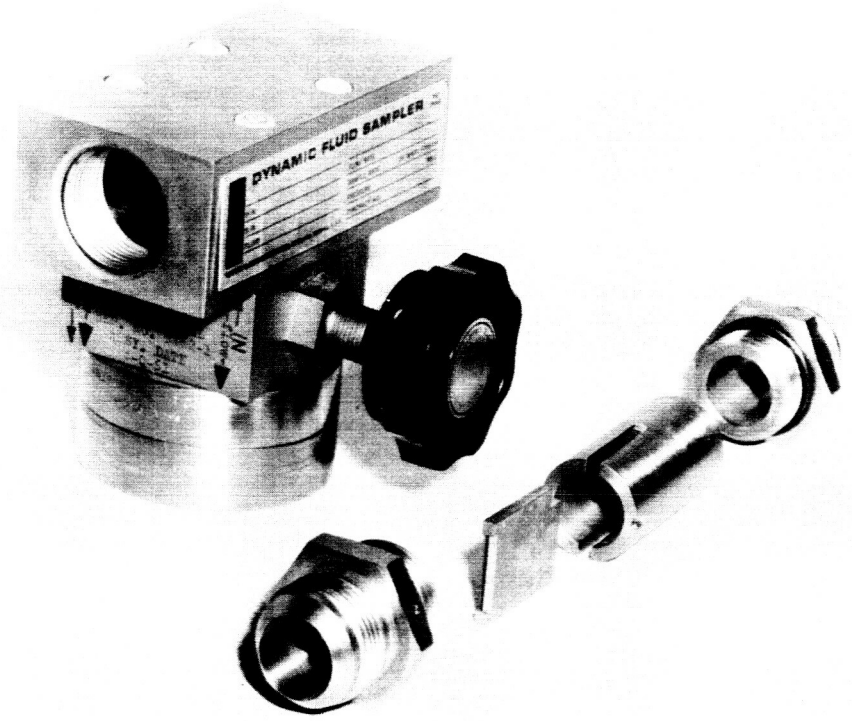



Figure 10. Wyle dynamic fluid sampler (partially exploded view)

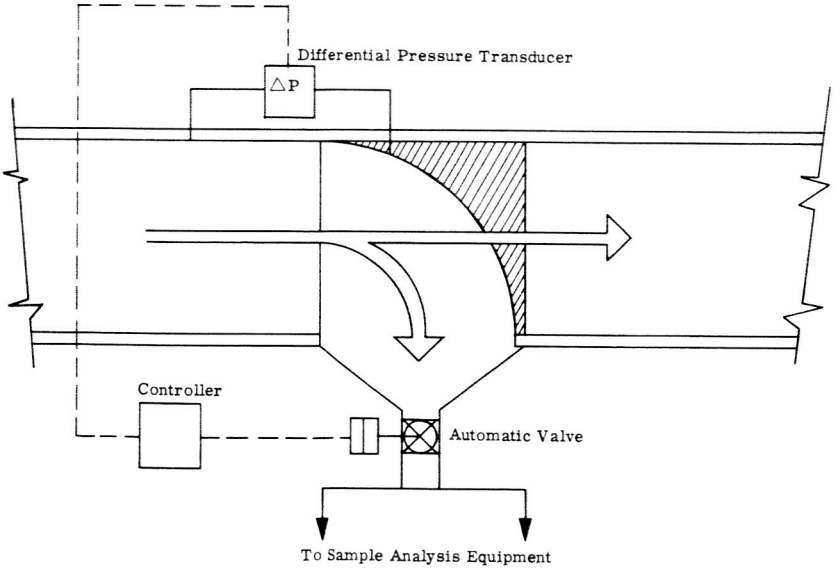



Figure 11. Dynamic fluid sampler installed for automatic inline sampling

In an operational system the dynamic fluid sampler would divert between 1 and 5 percent of the main fluid stream. For systems having a large flow rate, the diverted stream would flow through a second sampler, which would again divert 1 to 5 percent of the flow. Thus, a 1 gpm sample could be obtained from a system flowing 10,000 gpm.

## Purity Monitoring

### General

During the survey of launch and static test facilities only a few instances of on-stream monitoring of operational systems for the fluids of interest were found. These were:

1. Flame-ionization hydrocarbon analyzers for total hydrocarbons. These served both as continuous analyzers and periodic analyzers;

2. Moisture meters that read by the hydrolysis of absorbed water on hygroscopic plates. These are used for both continuous and periodic monitoring;

3. The fritted silver--KOH type $O_2$ analyzer for oxygen content in gases. These were used as continuous on-stream analyzers;

4. A gas chromatograph that was programmed by electronic tape for on-stream measurement of trace gas contaminants in hydrogen;

5. An instrument designed to measure the ortho-para ratio of hydrogen continuously on-stream;

6. A liquid process moisture meter that measured moisture content in petroleum products. This device was used continuously on-stream.

All of the organizations visited used various means of instrumental analysis in a laboratory for some contamination parameters in all of the fluids of interest, with the exception of $N_2O_4$. However, a number of these organizations still depended quite heavily on wet chemical analysis. In general, laboratory methods of instrumental analysis consisted of gas chromatography; infrared, ultraviolet, visible, and mass spectroscopy; electrolytic-type hygrometers for moisture content; and flame ionization meters for total hydrocarbon analysis.

Ideally, the ultimate contamination sensor would be a sensing probe connected to some type of readout device that indicates the amount of every contaminant in all fluids. Such a device does not exist and it is doubtful that it can be developed in the foreseeable future. Nevertheless a survey of instrumentation manufacturers was made to find available instrumentation capable of providing continuous monitoring of one or more of the parameters of interest.

Because very few types of continuous monitors were found, an alternative approach was formed. This approach was to provide an integrated system of continuous monitors for parameters that could be so monitored, and rapid recycle periodic monitors for those parameters that are impossible to monitor continuously. This integrated system comes as close to the idealized concept as is possible. Even though the system is not totally continuous, it would provide a better confidence level with regard to the condition of the propellant or pressurant that is loaded on the vehicle than does the present method of batch sampling.

The primary design criteria for a contamination sensor to be placed in given fluid system are that the sensor be capable of detecting and quantifying all parameters defined by the applicable specification and, in doing so, demonstrate accuracy and repeatability. The sensor must perform its operation of detection and quantification on a continuous basis and response should be as rapid as possible. Portability is desirable; however, it is not a firm requirement. If the instrumentation is installed permanently, it must be capable of withstanding the environment existing during launch. In addition, the instrument must be explosion proof if it is to be used in an area where flammable vapors might exist. The instrument must be of minimum size and weight consistent with all the other requirements stated.

Obviously the only criterion firmly defined above is that of detecting and quantifying and purity/impurity parameters specified in the applicable specification. Based on information supplied from the instrumentation manufacturers, no single instrument is available that can measure all these parameters. However, there are instruments that can measure a number of them. These instruments can be grouped into two separate types of systems. One system uses mass spectrometers in conjunction with hydrocarbon analyzers, moisture monitors, and ppm oxygen analyzers for the analysis of the contaminants in the cryogenic liquids and the gases. The other system substitutes process stream gas chromatographs for the mass spectrometer to provide the same analyses. Both systems would use process gas chromatographs for the analysis of the storable propellants and RP-1.

Both systems are capable of analyzing impurities to levels prescribed in the applicable specifications. However, the mass spectrometer is not capable of providing the analysis required for acetylene content (0.25 ppm) in oxygen. It also does not accurately provide the analysis required for the combined oxygen and argon content in hydrogen and helium (1.0 ppm) or the analysis required for hydrogen content in helium (1.0 ppm). The sensitivity of the mass spectrometer given for those analyses is $1 \pm 1/2$ ppm. This number indicates an error of $\pm 50\%$ at 1 ppm.

The manufacturers claim that the gas chromatograph is capable of making all the desired resolutions including the low ppm resolutions. However, it still is doubtful that any instrument can come any closer to these limits than the mass spectrometer. The moisture monitor, the ppm oxygen analyzer, and the hydrocarbon analyzer are capable of providing continuous monitoring at the desired levels of sensitivity. Therefore, these instruments can be used in most cases where these analyses are required. The mass spectrometer is not capable of continuous readout, but it is capable of performing specific analyses for individual

contaminants in approximately 1 second. The gas chromatographs take 15 seconds to 7 minutes to provide the same analysis. A 5-to-7 minute time span for analysis is the best that can be obtained by commercially available equipment. With considerable development of such new GC techniques as capillary column chromatography, the 5-to-7 minute time span can be cut to as low as 15 seconds, although 2 minutes appear more likely of attainment. Because of the extensive development requirement, it is impossible to establish a firm design criteria for time of analysis.

Size and weight of the moisture monitor, hydrocarbon analyzer, and the oxygen analyzer are well defined for the laboratory instrument. These are small, lightweight, portable instruments. The requirement for explosion and shock proofing these instruments will substantially increase their weight and size. This value cannot be defined for most instruments. The size and weight of the gas chromatographs are generally undefined because these parameters are a function of the technique used, e.g., a capillary-type chromatograph would weigh less than the conventional type.

No specific requirements for response time, accuracy, or repeatability can be given at this time for the gas chromatograph or the mass spectrometer because these parameters are also a function of developmental effort. Response times, accuracy, and repeatability for the total-hydrocarbon analyzer, the electrolytic hygrometer, and the infrared, ultraviolet, and oxygen analyzers are well defined.

Based on the previous discussion the following types of monitors are recommended for monitoring specific contaminants in the fluids pertinent to this discussion.

Gaseous Nitrogen

1. Purity can be monitored by instrumental determination of impurities and the automatic subtraction of these from the total;

2. Hydrocarbons can be monitored by a flame-ionization total-hydrocarbon analyzer;

3. Moisture can be monitored by an electrolytic hygrometer that indicates hydrolysis of absorbed water in hygroscopic plates or by a mass spectographic analyzer;

4. Trace $O_2$ can be monitored by a ppm oxygen analyzer, the style that has lead and fritted silver electrodes immersed in a KOH bath.

Helium

1. Purity can be monitored by instrumental determination of impurities and the automatic subtraction of these from the total;

2. $H_2$, $N_2$, $O_2$, and moisture can be monitored by a mass spectrographic analyzer;

3. Alternatives - $N_2$ and $H_2$ can be monitored by a process stream gas chromatograph; $O_2$ can be monitored by a ppm oxygen analyzer; moisture can be monitored by an electrolytic hygrometer;

4. Hydrocarbons can be monitored by a flame-ionization total-hydrocarbon analyzer.

Gaseous Oxygen

1. Purity can be monitored by the paramagnetic oxygen analyzer;

2. Moisture can be monitored by an electrolytic hygrometer;

3. Total hydrocarbons can be monitored by a flame-ionization total-hydrocarbon analyzer;

4. Acetylene content can be determined by nondispersive infrared spectroscopy. (It is not necessary to monitor for acetylene content in gaseous oxygen when total hydrocarbons are monitored. Acetylene is always monitored in $LO_2$, even though $LO_2$ is analyzed in the vapor phase.

Gaseous Hydrogen

1. Purity can be monitored by instrumental determination of impurities and the automatic subtraction of these from the total;

2. $O_2$, Ar, helium, carbon bearing gases, and moisture can be monitored by a mass spectrographic analyzer;

3. Alternative - These commodities can be measured by a process stream gas chromatograph;

4. Total hydrocarbons can be monitored by a flame-ionization total-hydrocarbon analyzer.

Liquid Nitrogen

No monitoring devices are capable of measuring contamination directly in cryogenic fluids. There is very little hope that such a device will be or can be developed in the future. However, for $LN_2$ and $LO_2$ of propellant grade, a continuous cryogenic sampler that uses a continuous flash vaporizer can be used in conjunction with the instrumentation proposed for the gaseous phase of these commodities to perform the required analysis. These cryogenic samplers are commercially available in several forms.

Liquid Hydrogen

For $LH_2$, a continuous cryogenic sampler, in conjunction with the instrumentation proposed to monitor $GH_2$, will be sufficient to analyze all contaminants.

A-50

The $N_2H_4$, UDMH, amine, and moisture content of A-50 can be monitored by a process stream gas chromatograph.

MMH

Purity and moisture content can be monitored by a process stream gas chromatograph.

$N_2O_4$

There is no known way of measuring purity or contamination content by instrumental methods. Some manufacturers have proposed the use of a process stream gas chromatograph for these analyses. However, each proposed chromatograph requires an extensive developmental effort, with no guarantee that the method will prove to be really satisfactory.

RP-1

1. Mercaptans can be monitored by nondispersive infrared at 3.92 $\mu$;
2. Aromatics can be monitored by nondispersive infrared at 5.1 $\mu$;
3. Olefins can be monitored by nondispersive infrared at 11 $\mu$;
4. Existing and potential gums cannot be monitored;
5. Moisture can be monitored by an ultraviolet analyzer;
6. All the above instruments require a vaporized sample for proper operation; therefore, these instruments must be used in conjunction with a high-temperature vaporizer and the analyses made in the vapor phase;
7. Sulfur in solution could possibly be determined by variations in light transmission monitored by a photocell. This technique will take a long time for development. Sulfur not in solution can probably be filtered out mechanically and will not contribute significantly to the total sulfur content.

Figure 12 shows a schematic of one of the liquid oxygen storage and transfer systems at Cape Kennedy, with suggested points for monitoring contamination. Suggested sampling points are indicated by S1, S2, etc.; suggested monitoring points are indicated by M1, M2, etc. $LO_2$ is received in transport trailers and transferred to the 900,000 gal. dewar located 1450 feet from the launch pad. A circulation pump could be installed between the fill line to the dewar and one of the main transfer lines to the S-IC to obtain tank samples (point S1) while $LO_2$ is circulated through valves A1 and A79. Point S1 can become a monitoring point by sharing equipment provided for M1 and M2 and the installation of switching equipment. The locations of monitoring points M1, M2, M3, and M4 are such that they are downstream from pumps and filters in the storage area and on the main fill and replenishment lines. Alternative monitoring points MA1 through MA5 are suggested for points that are nearer the interface of the vehicle and downstream of the final filter. However, these points are less desirable because of the environment the instruments will be exposed to and problems associated with sharing equipment. Optional monitoring points MO1 through MO3 are indicated in the event it becomes necessary to monitor the properties of fluid being returned to storage.

Point M1, on the main fill line immediately downstream of the storage area, would be equipped with a flame-ionization hydrocarbon analyzer, a moisture monitor, and a paramagnetic oxygen analyzer, to provide an indication of hydrocarbon content, moisture, and oxygen purity. An automatic particle counter, when available for liquid oxygen use, should also be located at this point.

Point M2, on the topping line in the storage area, would be equipped similarly to point M1. Point M3, on the topping line at the launch area and point M4, on the main fill line at the launch area, would be equipped with a hydrocarbon analyzer, a moisture monitor, and a particle counter.

Measurements from all points would ultimately be fed to the Launch Control Center so that all parameters would be monitored during loading. In the event that excessive contamination were detected during the loading or topping operations by the data processing system, automatic shutdown of the flow would be initiated, thus obviating the introduction of contamination into the airborne vehicle.

Similar monitoring systems for the other fluids could be set up in much the same manner. For all of the fluids there is available a good system of instrumentation consistent with the state-of-the-art that can provide continuous monitoring of contamination. Some of these instruments are off-the-shelf items while others will require some development. Nevertheless, the automation of contamination monitoring of operational fluid systems associated with large rocket boosters and spacecraft is well within the state-of-the-art; the implementation of automated monitoring of these systems is long overdue.

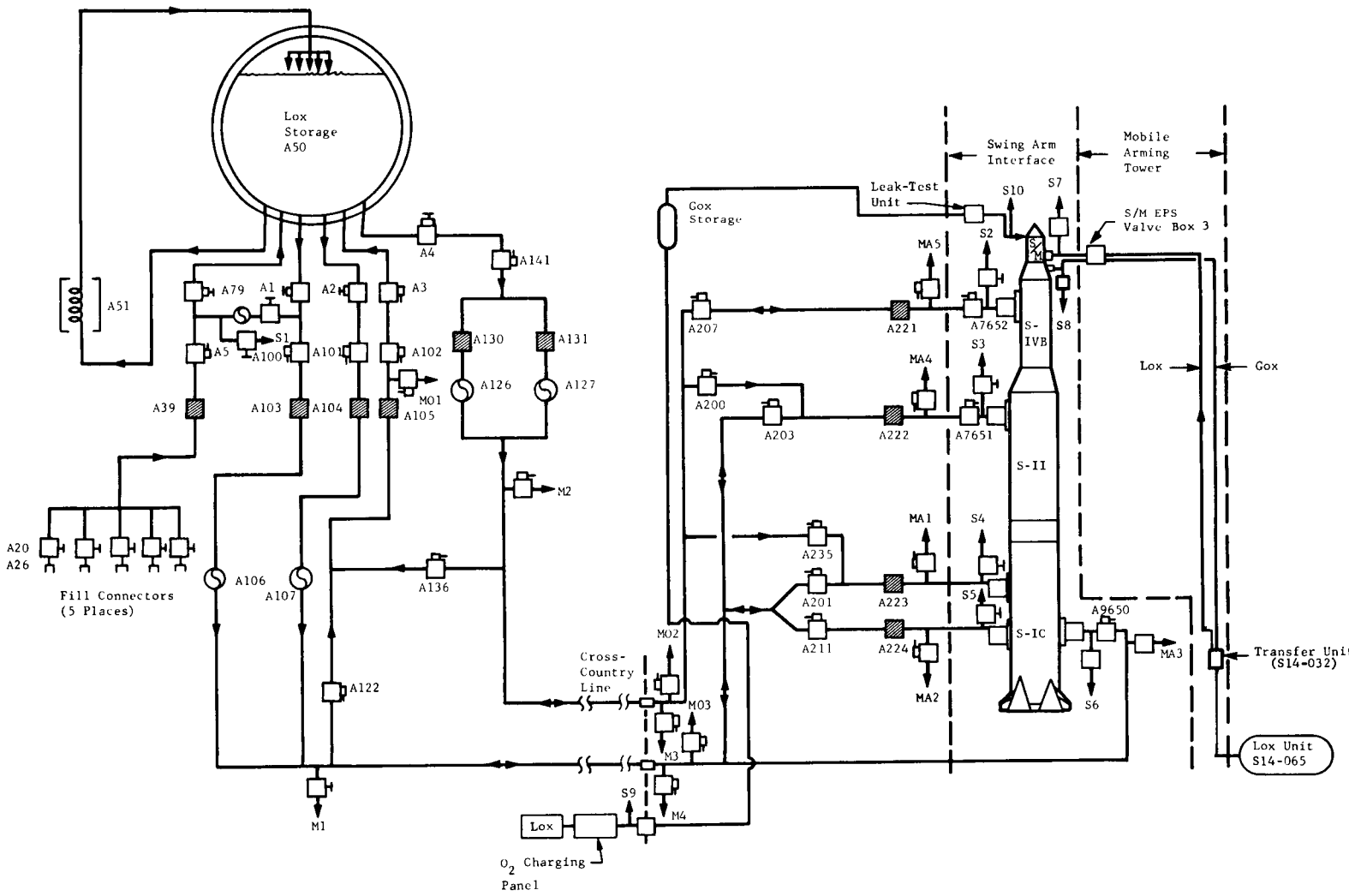



Figure 12. LC 39 $LO_2$ transfer system

# 5. MEASUREMENT OF CONDUCTIVITY OF LIQUIDS AS A MONITOR FOR CONTAMINATION

by

Sidney Balsbaugh, Balsbaugh Laboratories, Inc.,
and Verity C. Smith

Presented by

VERITY C. SMITH

Barnstead Still and Sterilizer Co. Inc.,
West Roxbury, Massachusetts

The problem of developing methods of measuring various impurities in liquids, gases, and solids, as well as the detection of contaminants on surfaces, has been of interest to the electronics, space, and atomic energy industries for many years; however, the technologies have become more sophisticated, and the requirements for cleanliness have also become more stringent. The method of test for traces of contaminants in materials used in production has been lacking so that production personnel have relied upon techniques of handling the materials as insurance against their contamination. If this had not been the case, progress in all of these fields would probably be in a sad state. On the other hand, there is no reason why we should not continue to work on improved test procedures for the detection of trace impurities, especially in the more critical areas. Solvents, both polar and non-polar, certainly fall into this category.

It is somewhat ridiculous to prepare a semiconductor device, or even the water system of the Apollo spacecraft, with all types of air filtering devices, pure gases, solutions of chemicals, and water if, as a final rinse, one uses an alcohol badly contaminated, not only with particulate matter, but also with dissolved ionized or un-ionized solids. Fortunately, particulate matter in the range of from 1/10th of a micron to 1 micron is coming under scrutiny as a result of the development of solvent resistance membrane filters. Of course, conventional membrane filters may be used for the study of particulate matter down to the range of 10 millimicrons in the case of liquids that do not attack the membranes.

Dissolved organic matter has come under investigation with the development of chromotography and infrared spectroscopy; however, these procedures are complex and, generally speaking, cannot be used on a continuous production basis. The industry obviously needs a quick, easy method to give the operator an idea of the relative purity of many solvents besides water. With this problem in mind, Balsbaugh and Smith

presented a paper at the ASTM Symposium, in October, 1962.[1] An attempt was made at that time to establish values of resistance of Freon®, trichloroethylene and isopropyl alcohol measured at various temperatures at a purity which was hoped to be close to the ultimate for practical purposes. The data clearly indicated a drop in the resistance with an increase in temperature, which seemed to be quite consistant.

Since that time the subcommittee on Contaminants in Liquids of ASTM Committee F-1 has drawn up a procedure for measuring the resistance of solvents, Method F-58-65T. Because there has been little quantitative data available on the relationship between the resistance of the solvent and the actual purity, the method has not been practiced to any extent as far as we know, though it appears that further consideration should be given to this method of approach.

For this symposium, Mr. Balsbaugh and Mr. Smith set up essentially the same distillation apparatus used previously in an attempt to prepare a relatively pure solvent which could then be measured before and after being contaminated by various impurities. Time was limited so that the complete investigation of contamination, not only by solid surfaces, but also by gases and liquids was impossible, and concentration was on detecting the change in resistance of the solvents before and after they had been in contact with copper, stainless steel, and Teflon®.

The distillation apparatus consisted of a Barnstead model EPR-1/2 Quartz Redistiller followed by a guarded Balsbaugh type 100T3 Conductivity cell designed to measure the resistance directly from the distillate column. Following this cell the solvent was passed through a piece of block tin tubing immersed in a constant temperature water bath. Subsequent to this, we had a Pyrex® cell holder containing cell No. 2, then the test coil immersed in the same constant temperature bath followed by cell holder No. 3 and cell No. 3 of the same design. All measurements on the Freon were made on a General Radio type 1230-A Electrometer. This standard dc amplifier and electrometer was used with a 90-volt auxiliary battery supply. The isopropyl alcohol was measured with a Balsbaugh Laboratory Bridge, Model 100LRB.

We ran the tests at different temperatures and attempted to hold the flow rate constant through the copper, stainless, and Teflon coils. The tin coil was used merely to reduce the temperature of cell No. 2 to the temperature of the outlet cell. We recognized that the tin might contaminate the liquid. However, it was chosen on the basis of its general passivity toward water and also a verbal communication with an engineer at the Dupont Freon Products Division indicated that tin sealed inside a glass tube in contact with Freon seemed to have been less affected by the Freon than any of the other metals tested.

It was hoped that we would find a relationship between the resistance and the contact time with the various sections of tubing. We had also hoped that there might be some temperature relationship,

---

[1] Fourth Pacific Area Meeting of ASTM, Los Angeles, California, October 1-3, 1962. This paper "Cleaning and Materials Processing for Electronics and Space Apparatus" is found in the ASTM Special Technical Publication No. 342 and is obtainable from the Society Headquarters.

though the mechanism of contamination of a nonpolar solvent by a solid is not necessarily temperature sensitive, especially in the short range from 280° to 320°K. Analysis of the assumed contaminated solvents was also conducted to try to obtain quantitative information.

Fortunately, we did get a clear indication in all cases of a drop in resistance with the passage of the liquid through the tubing. These data are shown in Table I for Freon and isopropyl alcohol in contact with Teflon, copper, and stainless steel tubing. The tubing in each case had an approximate inside area of 1 square foot, which gives a constant of 0.157 milliliters per square centimeter per second when the flow rate of the liquid through the apparatus is considered. The table has columns on the change in resistance and on the percent difference in the resistance reading before and after the contaminating coil. We felt this was more realistic because the numbers are large, in the range of $10^{16}$ for Freon and $10^{7}$ for isopropyl alcohol; thus, the pure change in resistance is not too significant.

TABLE I

Change in Specific Resistance of Freon and Isopropyl Alcohol after Contact with Copper, Teflon and Stainless Steel at 25°C

FREON

| *Coil Material* | *$\triangle R$* | *% Change* |
|---|---|---|
| Teflon | $30.78 \times 10^{15}$ | 51.4 |
| Copper | $19.47 \times 10^{15}$ | 63.0 |
| Stainless Steel | $15.47 \times 10^{15}$ | 54.7 |

ISOPROPYL ALCOHOL

| | | |
|---|---|---|
| Teflon | $0.97 \times 10^{7}$ | 8.3 |
| Copper | $3.57 \times 10^{7}$ | 43.5 |
| Stainless Steel | $3.55 \times 10^{7}$ | 49.4 |

It is interesting to note that with the exception of isopropyl alcohol passing through Teflon, the relative change in the resistance approached 50 percent in the case of both Freon and isopropyl alcohol. It also may be significant to report that when we compared the results of the previous work with these data, we find that we assumed pure Freon had a resistance of approximately $3.4 \times 10^{16}$ ohm-cm at 25°C; we find that the Freon at No. 2 cell under the same condition has a resistance of $3.1 \times 10^{16}$. This resistance dropped to $1.1 \times 10^{16}$ after passage through the copper coil, $1.3 \times 10^{16}$ after passage through the stainless steel coil, and $1.4 \times 10^{16}$ after passage through the Teflon coil. The temperature variation did not seem to have a significant effect, but this may have to be studied further.

When these experiments were conducted, an attempt was made to test for copper in the solvent that had been in contact with the copper coil, and iron in the solvent that had been in contact with the stainless steel coil. Though the method used for copper should have been sensitive in the range of a fraction of a part per billion, we found no indication of any copper. Likewise, we found no detectable iron. These colormetric methods of detection of ions certainly cannot be considered too good if we are concerned with molecular contamination in the true sense.

An examination of the electrical characteristics of a single electron might be considered at this point, since there may be considerable significance to the electrical charge of a single ion and a quantitative deduction on the purity of the solvent. If it is assumed that the charge on the electron is $4.8 \times 10^{-10}$ electrostatic units, one may convert this to $1.58 \times 10^{-19}$ amp-seconds per electron. If we go one step further, we can conclude that a solvent having a resistance of $10^{16}$ ohm-cm, measured at a voltage of $10^{-3}$ would have an amperage of $10^{-19}$, which is approximately equivalent to 1 electron. Likewise a resistance of $10^{16}$ with a voltage of 1/10th would have an amperage of $10^{-17}$, roughly equivalent to 60 electrons. It is quite possible, therefore, that we are actually measuring molecules of impurities in the Freon which would be completely undetected by other techniques.

We are also aware of the fact that a polar solvent, such as isopropyl alcohol with a measured resistance in the range of $10^{7}$ would, by its nature, have a measurement of approximately $10^{10}$ molecules because of the inherent disassociation of the isopropyl alcohol molecules. We also note that the isopropyl alcohol shows a considerable drop in resistance with passage through the two metals, but not through the Teflon, indicating that the impurities picked up from the Teflon are unimportant when compared to the number of molecules already disassociated in the liquid.

Being aware of the pitfalls involved in measuring very high resistances, we considered the possibility that this resistance might be altered merely by the streaming potential of the liquid passing through the tubing. On the other hand, our previous work showed that the resistance did not change appreciably with a change in flow rate over a fairly wide range, which tends to lessen our concern in this area. We also realize that particulate matter, when suspended in a nonconducting liquid, will set up a potential in this liquid. It is, therefore, possible that particulate matter might be responsible for the apparent change in resistance. Similarly, molecules of organic matter dissolved in the solvent might change the resistance merely by their presence in the electrical field of the cell.

Though we do not feel that we have proved that the resistance of the solvents tested varies quantitatively with any particular impurity, we do feel that it is safe to assume that solvents in contact with a solid will become contaminated by that solid to a degree. We also feel that this contamination can be measured by measuring the resistance of the solvent, and that the users of the various solvents can quite easily set up standards based on resistance measurements.

It would be the responsibility of each process engineer to develop his own standards based on the practical recognition of the fact that one must be able to produce the solvent of the standard required at the site, or one must have a method of transporting it to the point of use in the uncontaminated state. Our informal check on the resistance of purchased, high grade solvents as obtained from the container, indicate that the conductivity varies widely from batch to batch and that most of them are quite contaminated if the comparison between the conductivity of freshly distilled solvent is made with that same liquid after it has been stored in a container. We strongly suggest that one should not overlook this relatively inexpensive, simple approach to the detection of contaminants in solvents.

# 6. CONTAMINATION EFFECTS AND CONTROLS IN SATURN LAUNCH VEHICLE HYDRAULIC SYSTEMS

by

Victor R. Neiland

Presented by

F. J. BEYERLE

Propulsion and Vehicle Engineering Laboratory
Marshall Space Flight Center
Huntsville, Alabama

## Abstract

Unlike industrial or aircraft liquid systems, man-rated launch vehicle systems must always function to insure a successful and safe mission. The background and present concept relating to contamination control of SATURN hydraulic systems is presented. Contributors to contamination and contamination tolerance levels are discussed. The contamination control approach evolved and employed for the SATURN hydraulic systems is also presented.

## Introduction

Fluid system contamination became an acute problem during the development of a ballistic missile about 10 years ago. From then until now, the aerospace industry has accepted the contamination challenge and a decade later is finally able to understand and effectively control it.

Only retrospectively can one appreciate the progress made in this field during the past decade. Figure 1 shows a typical hydraulic system of 10 years ago. Ground equipment cleaned and purged the on-board hydraulic system. Since a number of features were "added on" to the system, the resulting network was difficult to purge. Many "dead ends" and restricting branches were present. It was not unusual for cleaning operations to last for 40 hours. Ground filtration of the low micron depth type was not generally available for medium pressure systems. The Whatman method of determining contamination levels did not give consistent results since some contamination became obscured within the sample patch.

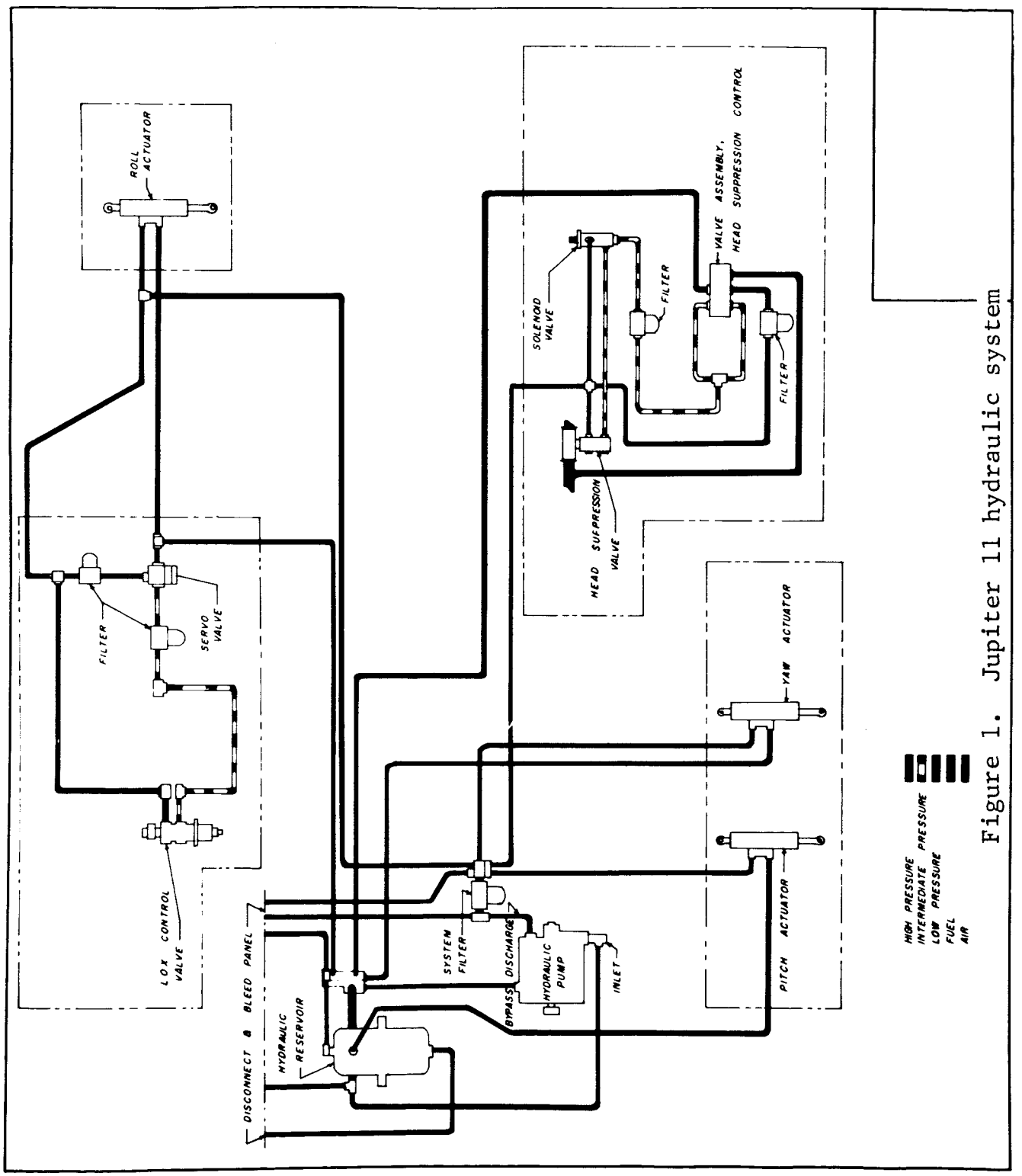



Figure 1. Jupiter 11 hydraulic system

Even though wire mesh filters were available, they were poorly designed from a structural standpoint and were capable of media migration. Other media were tried with poor results (see Figure 2).

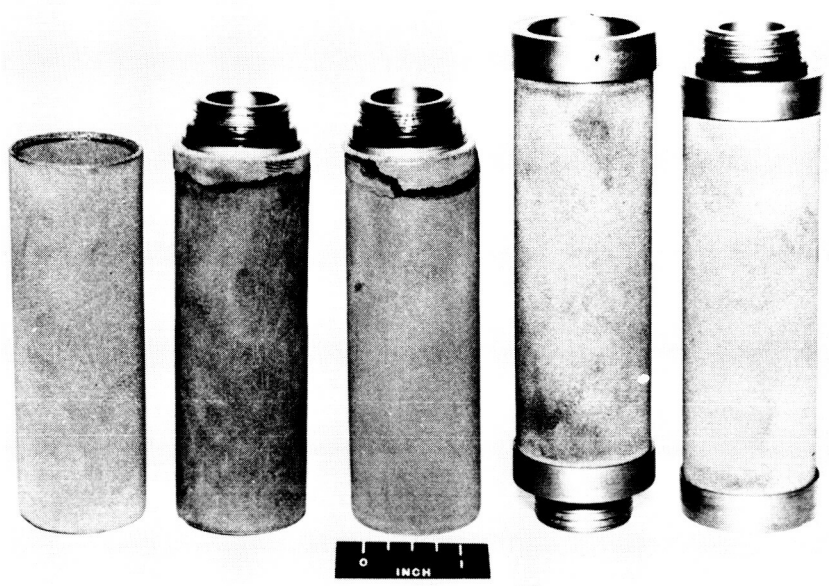



Figure 2. Failure of sintered bronze filter elements after vibration

Servo valves were very sensitive compared to components that are currently available. "Built-in" contamination was not unusual. Valves used for sampling the system added considerably to the system level. Sampling was done at manifolds far removed from the sensitive component and periodically "unloaded" contaminant so that the sample was not reliable.

The hydraulic industry was not able to aid the plight of the user, since little had been done to investigate this area. Component cleaning was considered time consuming and costly. Clean rooms were in their infancy. Hydraulic components were not readily available and uprating and modification of existing items were frequently required.

## Sources and Effects of Contamination

Contamination becomes a part of a fluid system in one of three ways:

1. Generated
2. Built-in
3. Environment

Generated contamination is formed by any dynamic mechanism, such as a pump, accumulator, or actuator. This material can be formed by wear, erosion, cavitation, or abrasion. The migration of filter media under certain environmental stimuli can generate contaminants. Another form of generated contamination is caused by the breakdown of the fluid or its interaction with other substances within the system. Illustrations of generated contamination can be seen in Figure 3.

"Built-in" contaminants can be controlled by better processing of components. Particles left over from manufacturing operations, such as machining chips and burrs, weld slag, lapping compound, scale, oxides, corrosion products, lubricants, waxes, residual fluids, and material introduced by ground units are sources for this type of contaminants. Examples of internal contamination are shown in Figure 4.

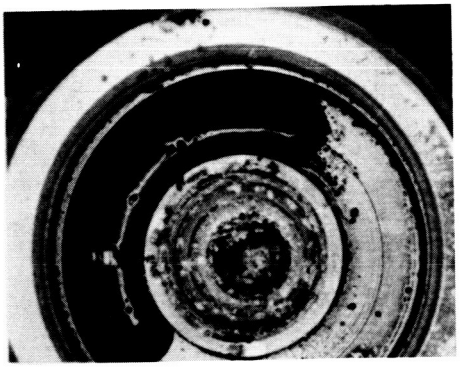
Contamination introduced from the outside environment when the system is serviced or actuated constitutes another source of contamination. During operation, material can enter through vents, filling ports, actuator pistons, or relief valves.

The last two types of contamination can be minimized by an effective program of component cleaning and system servicing in a controlled working area. Generated contaminant can be reduced by component design changes, material substitutions, protective finishes, careful acceptance tests, investigating abnormal conditions and taking corrective action.

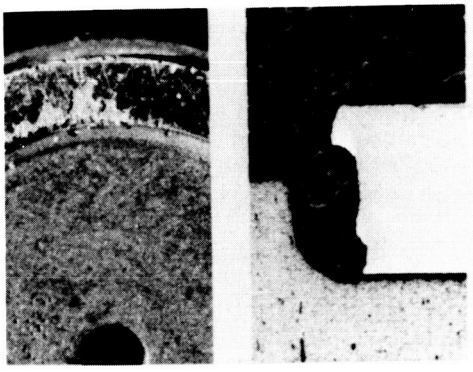
Thus, the contamination level of a dynamic fluid system is the degree of cleanliness at any specific time resulting from built-in contaminant, generated contaminant, environmental contaminant, and contaminant removed from the system by filtration (see Figure 5B).

The sensitivity or tolerance level of a system is determined by the ingredients that go to make up the system and its operational mode. These factors and the considerations associated with them are as follows:

1. Component configuration:
   - element design
   - clearances
   - surface finishes
2. Component materials:
   - seals
   - moving parts
   - bearings
   - housings and sleeves
3. Operational requirements:
   - speed
   - pressure
   - temperature
   - vibration, surge

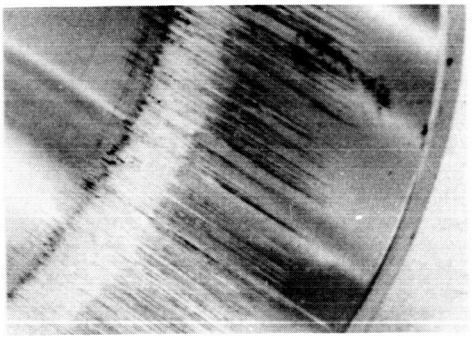


A. FILTER HOUSING CORROSION

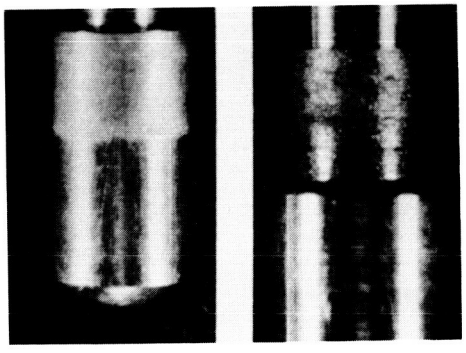


B. PUMP PISTON SHOE EROSION -CAVITATION

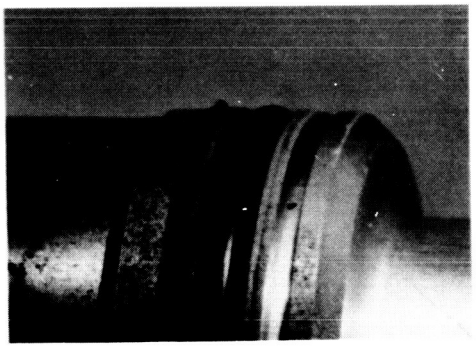
C. PUMP CYLINDER BLOCK CAVITATION - EROSION



D. ACCUMULATOR SLEEVE ABRASION

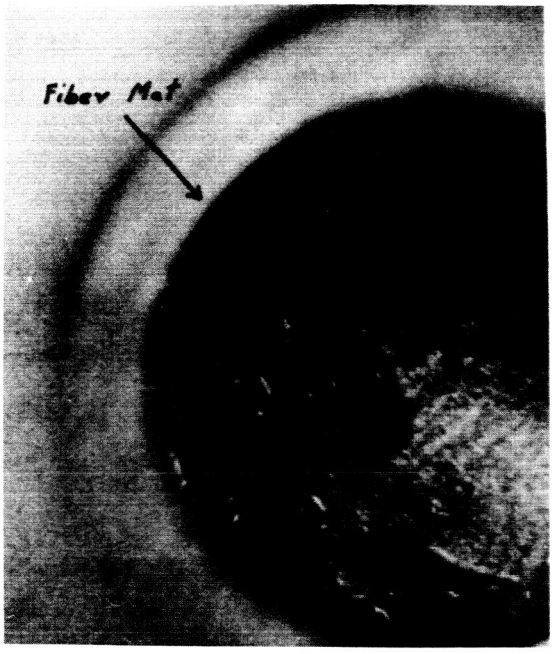


E. PUMP COMPENSATOR SPOOL EROSION

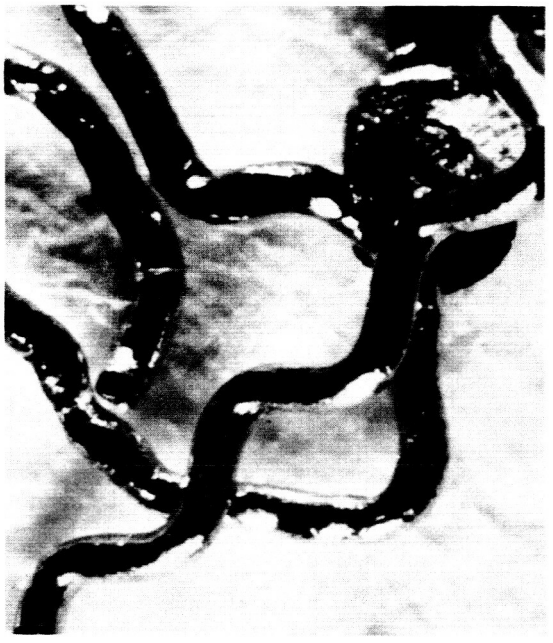


F. RESERVOIR PISTION O-RING WEAR

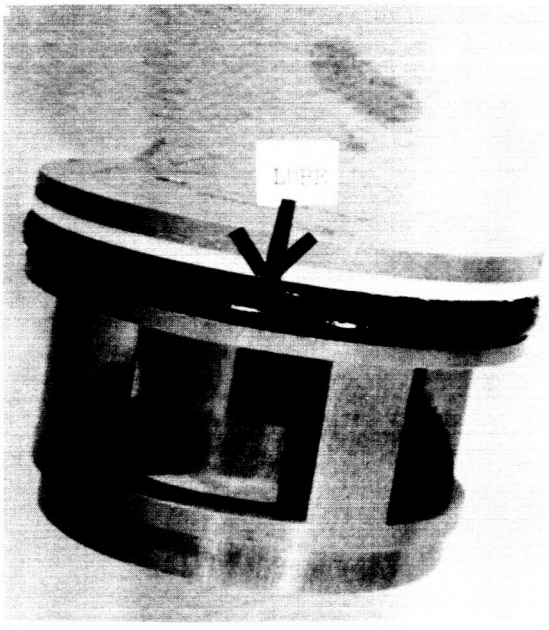
Figure 3. Generated contamination



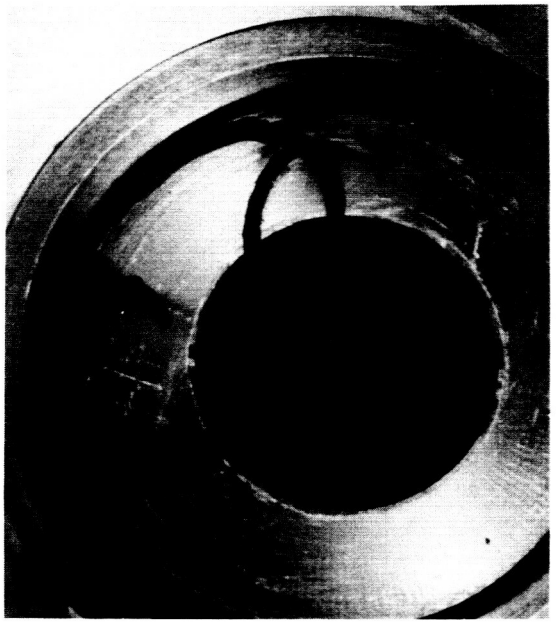

A. FIBER MAT CAUSING RELIEF VALVE TO FAIL OPEN



B. WIRES FOUND DOWNSTREAM OF WIRE MESH FILTER ELEMENT



C. LUBRICANT IN FILTER MANIFOLD



D. CUT O-RING IN FILTER ELEMENT CAVITY

Figure 4. Built-in contamination

4. Fluid properties:
   - viscosity
   - film strength
   - surface tension

These factors define the physical limitations of a hydraulic system and will determine how sensitive the system will be and what contamination controls are required for satisfactory and reliable operation (see Figure 5A).

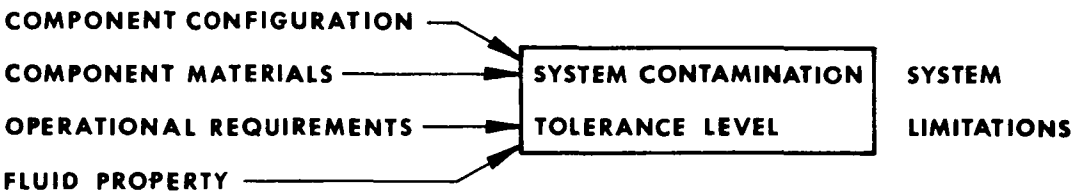



Figure 5A. Contributors to the system contaminant tolerance level

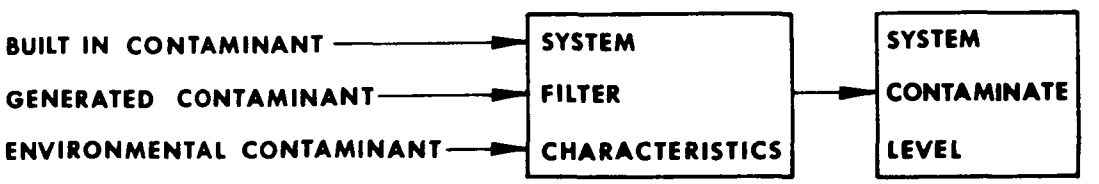



Figure 5B. Contributors to the system contamination level

Hydraulic pumps are generally considered one of the major contributors to contamination in a hydraulic system. Figure 6 shows a characteristic pump contaminant generation time curve. Pumps used for Saturn usually are subjected to 12 to 15 hours of testing during manufacture and acceptance. This places the pumps on the decreasing or lowest portion of the curve.

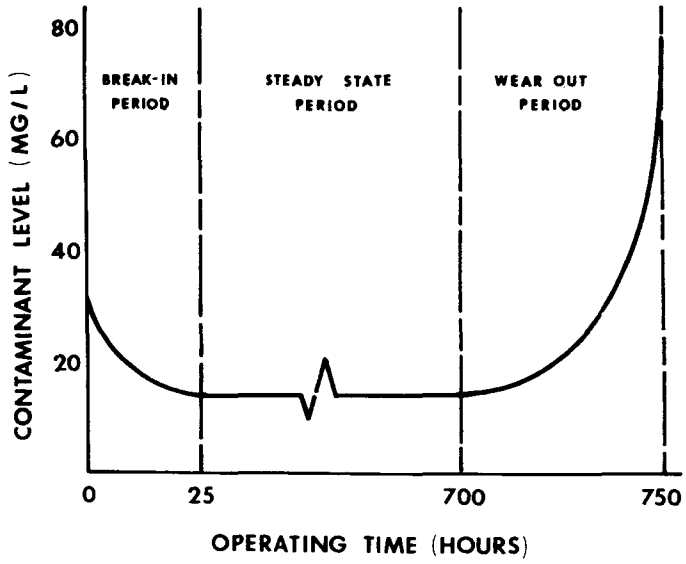



Figure 6. Contaminant generation periods for pumps

In most self-contained hydraulic systems, the large particles are usually well controlled by recirculating flow through filters. However, many areas in the system are vulnerable to particles below 5 microns in the form of silt, especially if they are metallic. Some typical clearances encountered in high performance hydraulic systems are shown in Figure 7A, and valve silting time versus fluid contaminant level in Figure 7B. Recent work done at Oklahoma State University[1] has shown that carbonyl iron can cause spools to stick more readily than AC fine dust. The dilution of the carbonyl iron contamination with AC dust will inhibit valve sticking. For silt to have a particularly damaging effect, it must be tuned to the critical spool clearance. Figure 8 shows a hydraulic pump piston which broke off from the rotating group because of sticking caused by contamination.

## Saturn Hydraulic Systems - Description

Figure 9 shows the three Saturn launch vehicle configurations. Saturn I is a two-stage vehicle that served as a test bed for the evolving Saturn family. An improved Saturn I, which contains a powerful S-IVB stage, is identified as the uprated Saturn I. Finally, Saturn V is a 3-stage vehicle designed to propel the Apollo modules to the moon. Figure 10 shows some of the salient features of this vehicle.

[1]Fluid Power and Controls Laboratory; Dr. E. C. Fitch - Director Study of Filtration Mechanics and Sampling Techniques; NAS8-11009.

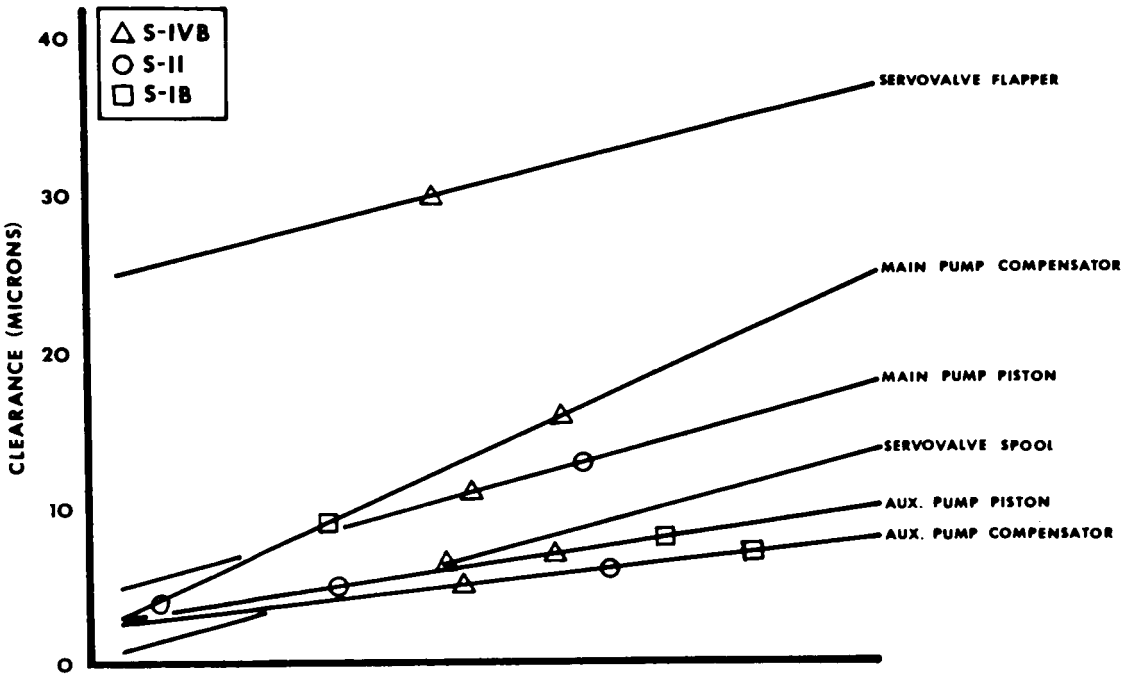



Figure 7A. Critical clearances in hydraulic system

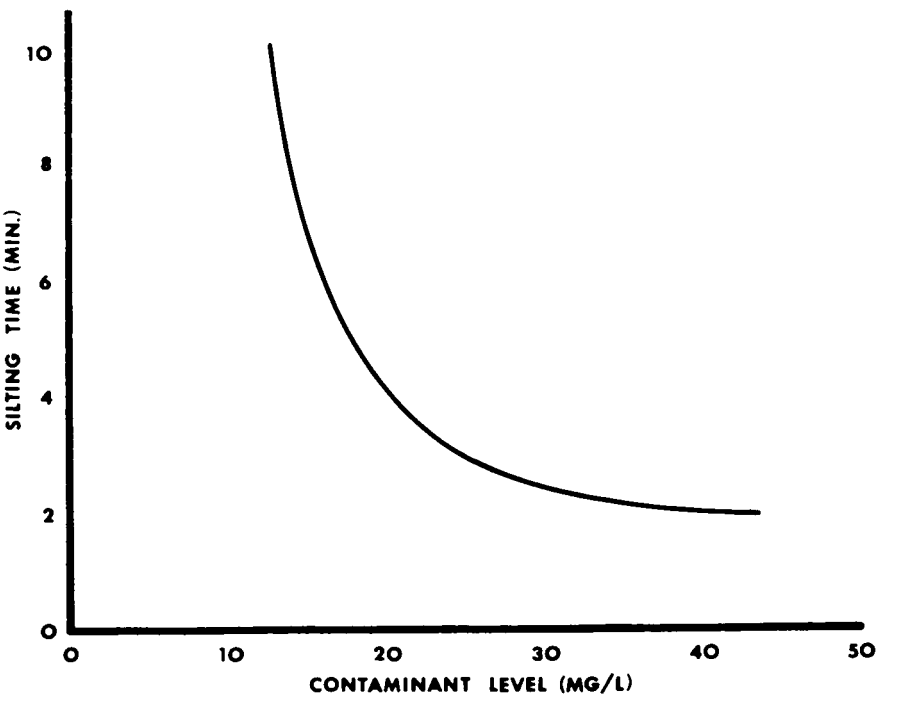



Figure 7B. Valve silting time versus fluid contaminant level

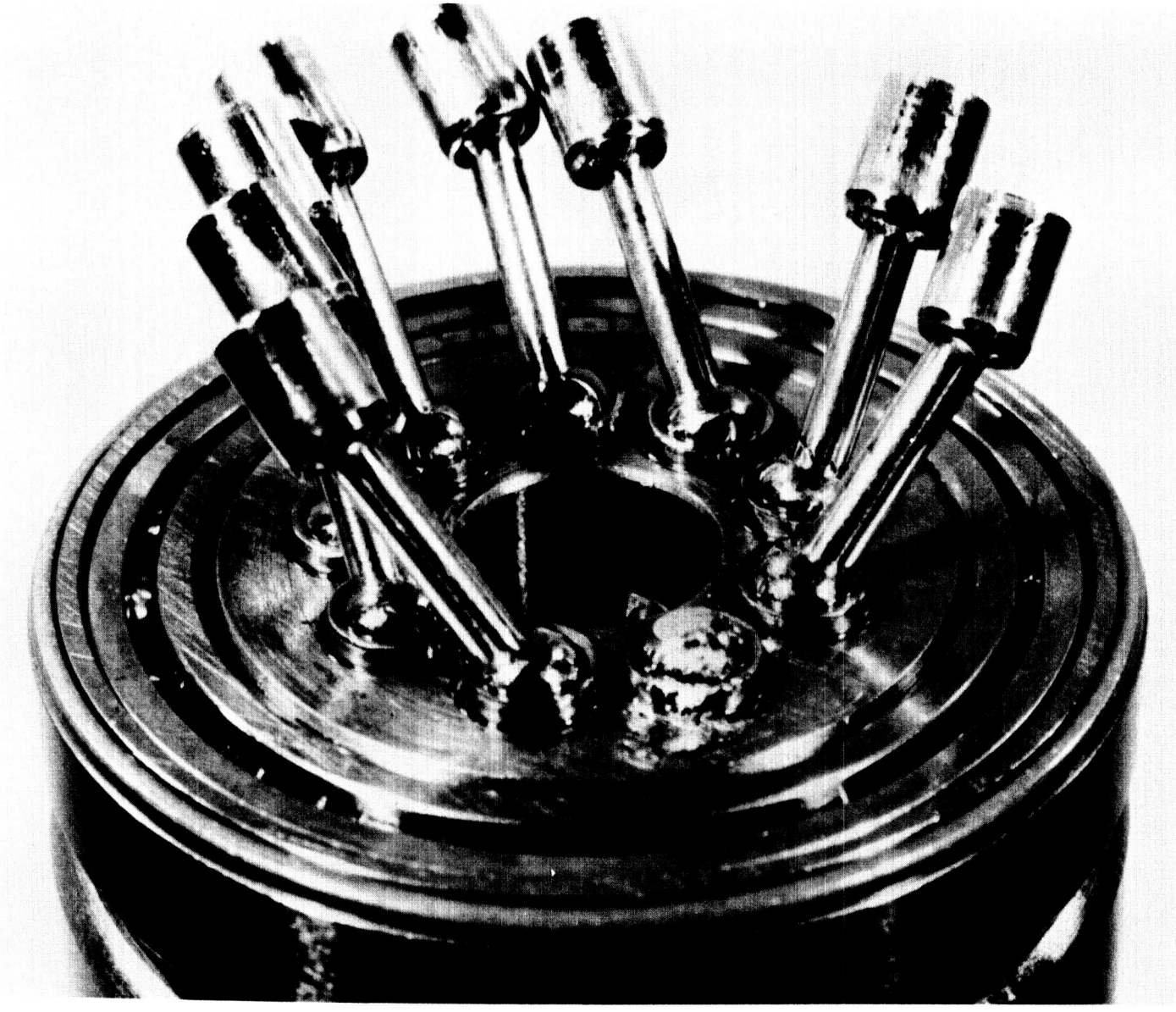


Figure 8. Hydraulic pump piston failure due to contamination

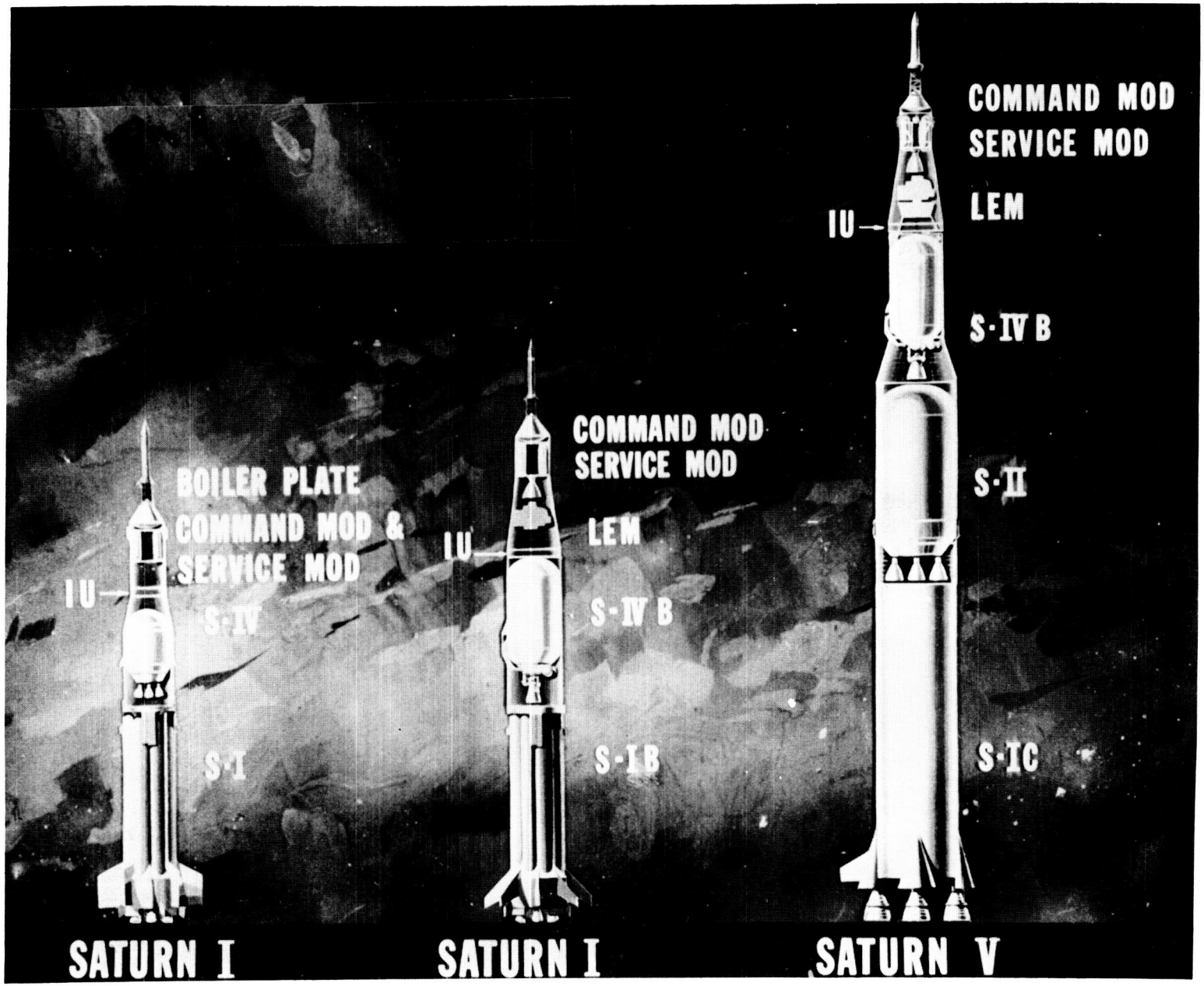



Figure 9. Saturn vehicles for Apollo

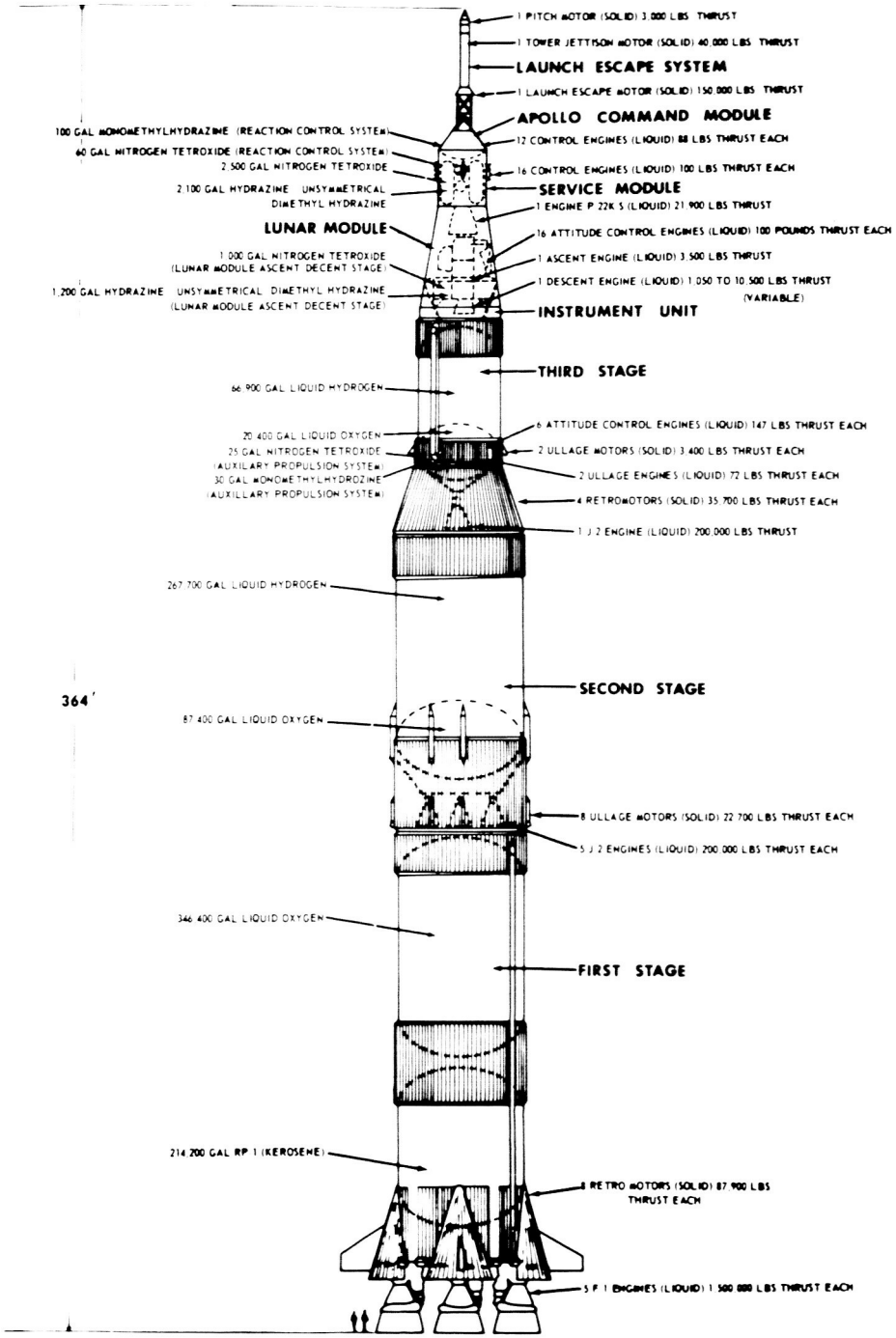



Figure 10. The Apollo/Saturn V

Saturn launch vehicle hydraulic systems furnish fluid power to gimbal vehicle engines for thrust vector control. An independent, closed loop system with built-in checkout capability is installed on or near each gimbaled engine of the S-IB, S-II and S-IVB stages. Each system consists of a main hydraulic pump (engine driven), an auxiliary hydraulic pump (electric motor driven), an accumulator-reservoir module, and two servoactuators. These components are interconnected by tubing and flexible hose assemblies. Figures 11, 12 and 13 illustrate these features.

The S-1C system initiated and developed by MSFC in 1962 departs somewhat from the closed-loop concept. At engine start, the engine turbopump accelerates, raising the fuel discharge pressure above the ground supply pressure and cycling the filter manifold check valves. During flight, fuel (RP-1) remains the source of fluid power to the servo actuators. This approach was taken for the following reasons:

1. Accessory power pad for hydraulic pump would add complexity and weight.
2. Ground system would be available to start rocket engines.
3. High pressure RP-1 fuel source was available (17-1900 psi) from the turbopump.
4. Accumulator-reservoir and associated valving is not required from a functional standpoint.

Until engine start, ground supply fluid passes through the filter manifold and to the servo actuators. This system consists of two servoactuators, an upstream filter manifold, and connecting supply and return ducting.

## Contamination Control of Saturn Hydraulic Systems

The new generation of hydraulic systems attempted to minimize the contamination problem by using a many faceted approach. Possibly the key and most important contribution to the problem was the development and industry wide implementation of the SAE ARP 598 particle counting method, which provided a reference standard technique in addition to an absolute method for determining the degree of system contamination.

"Built-in" contaminant is greatly minimized by cleaning of all components before assembly in accordance with specification MSFC-PROC-166, or its equivalent (see Table I). System levels for Saturn hydraulic systems are also specified (see Table II).

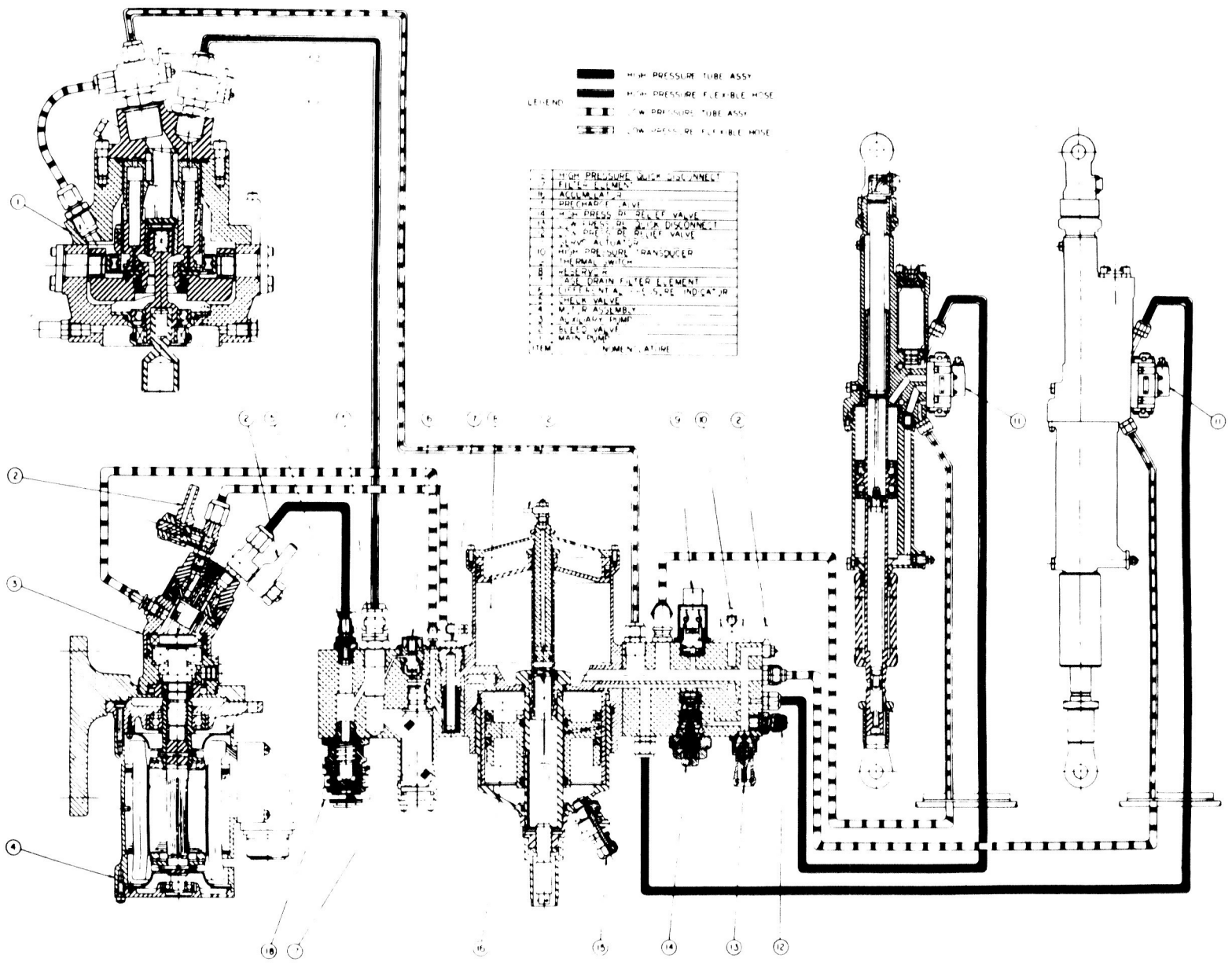



Figure 11. S-I hydraulic system schematic

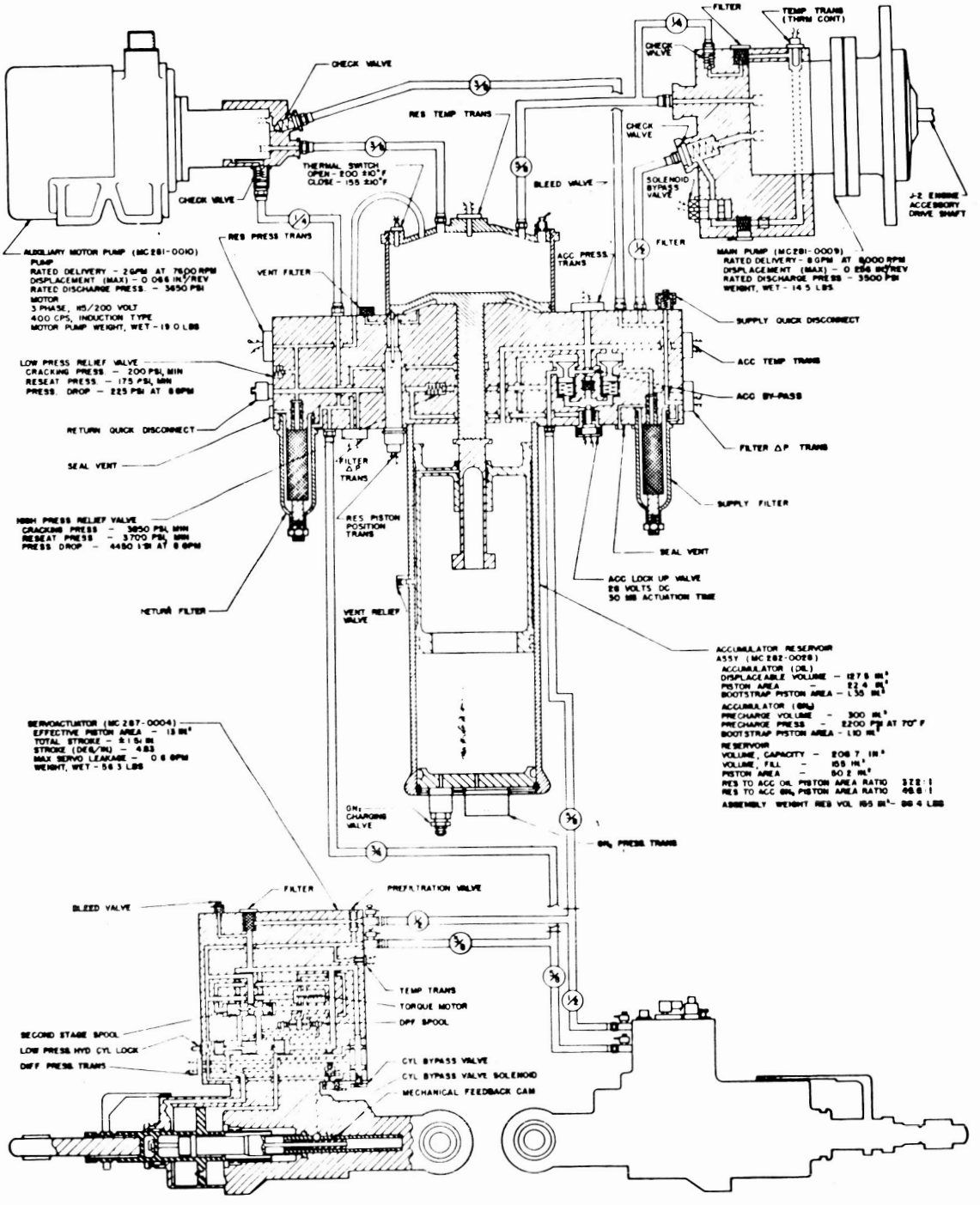



Figure 12. Engine gimbal system schematic

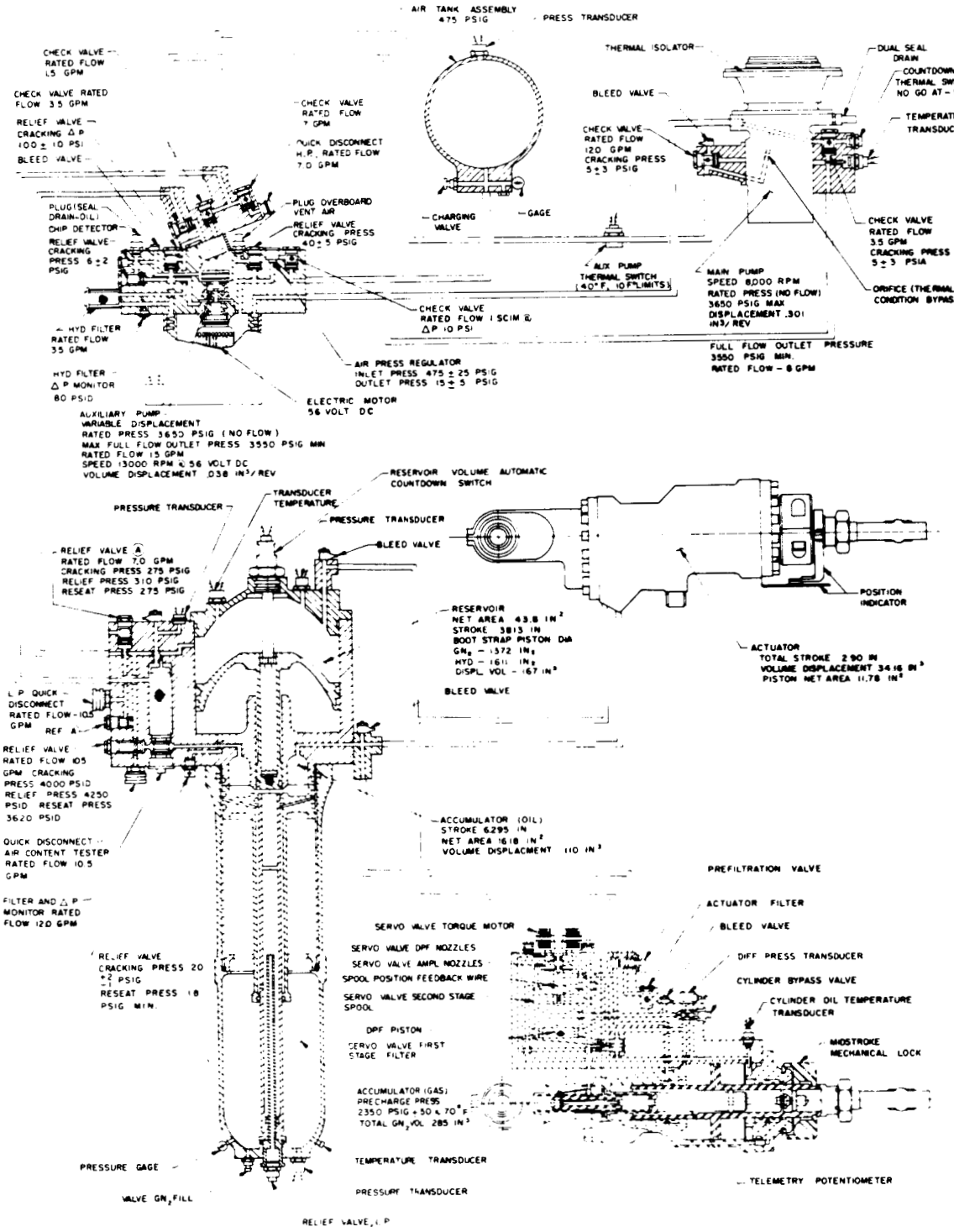



Figure 13. S-IVB engine gimbal system schematic

TABLE I

Contamination Limits

| ITEM | SAMPLE VOLUME | PARTICLE SIZE (MICRONS) | | | | SATURN | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 10 - 25 | 25 - 50 | 50 - 100 | OVER 100 + FIBERS | 1B | 1C | 11 | 1VB |
| 1. HYDRAULIC FLUID FOR TESTING ASSEMBLIES | 100 ML | 1,340 | 210 | 28 | 3 | X | | X | X |
| 2. FILTER ELEMENT | 2 LITERS | 50,000 | 1,250 | 100 | 10 | X | X | X | X |
| 3. COMPONENTS | 100ML/FT$^2$ | 600 | 100 | 16 | 2 | X | | | |
| (SAMPLE OF FINAL | 100ML | 300 | 50 | 8 | 1 | | | X | X |
| FLUSH FLUID) | 100ML/FT$^2$ | 900 | 150 | 40 | 4 | | X | | |
| 4. ASSEMBLIES IN TEST (EFFLUENT OIL) | 100 ML | 2,150 | 530 | 60 | 10 | X | | X | X |

TABLE II

Specification Limits for Hydraulic Systems

| ITEM | SAMPLE VOLUME | PARTICLE SIZE (MICRONS) | | | | SATURN | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | 10 - 25 | 25 - 50 | 50 - 100 | OVER 100 + FIBERS | 1B | 1C | 11 | 1VB |
| 1. SERVICER BEFORE CONNECTING TO VEHICLE SYSTEM | 100 ML | 1,340 | 210 | 28 | 3 | X | | X | X |
| 2. SUPPLY TO ONBOARD SYSTEM | 100 ML | 2,150 | 530 | 60 | 10 | X | | X | X |
| FLIGHT MODE | | 6,500 | 1,600 | 300 | 40 | | X | | |
| GROUND MODE | | 3,250 | 800 | 150 | 20 | | X | | |
| 3. RETURN FROM ONBOARD SYSTEM | 100 ML | 4,300 | 1,060 | 120 | 10 | X | | X | X |
| GROUND MODE | | 13,000 | 3,200 | 600 | 40 | | X | | |

Environmental contaminant is controlled by limiting ground systems to filling and replacement of key components. Operational check-outs are performed, using "on-board" components. Figure 14 shows the gradual reduction of operation time for the first 16 H-1 Engine Gimbal systems, during flushing and purging. "Open operation" refers to the time the prefiltration bypass valve prevents hydraulic oil from going through the servo valve, thereby preventing entrance of contamination that may have been picked up in other portions of the system. Closed operation is the normal mode with fluid flowing through the servo valve.

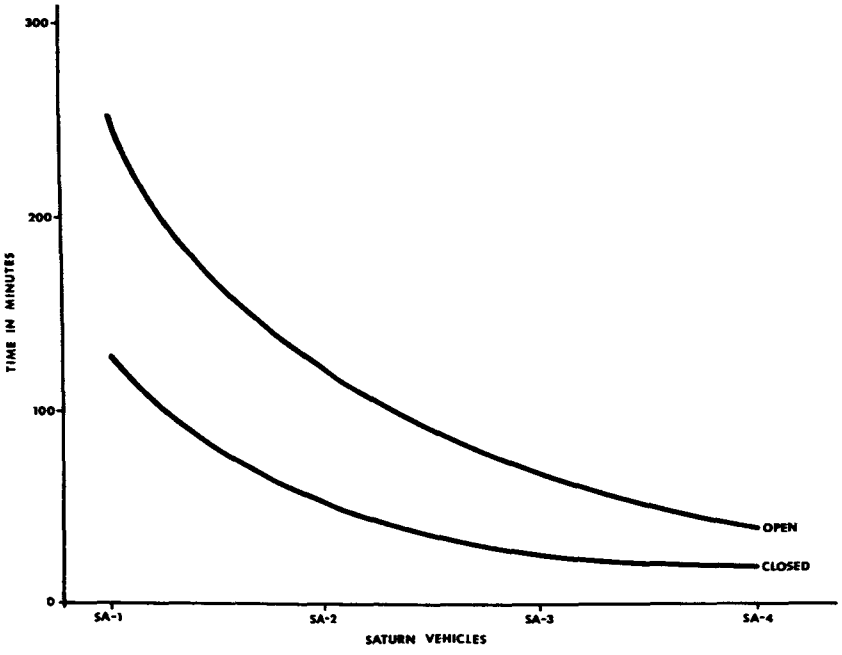



Figure 14. Average purging and flushing time for four Saturn hydraulic systems

Supercleaning of hydraulic fluid before it is incorporated into the ground system is an important consideration. Additional cleaning and circulation is accomplished within the servicer. High-capacity depth filters are employed. Another major consideration is greater scrutiny of hydraulic components during incoming and in-plant acceptance tests to verify acceptability from an operational and contamination standpoint.

Systems are tested at specified contamination levels for many hours to gain confidence that the system will reliably operate for the necessary minutes in flight. In addition, flight type hardware has undergone hundreds of hours of closed system operation. Pumps and filters have also been tested as components and subjected to flight environments. Figure 15 shows the composite vibration spectrums that the major components are subjected to. Filters have shown a performance degradation

when subjected to vibration and surge. The housing can become a major generator of contamination when it is undergoing flight vibration. To account for this added influence on contamination level, conservative system levels are specified.

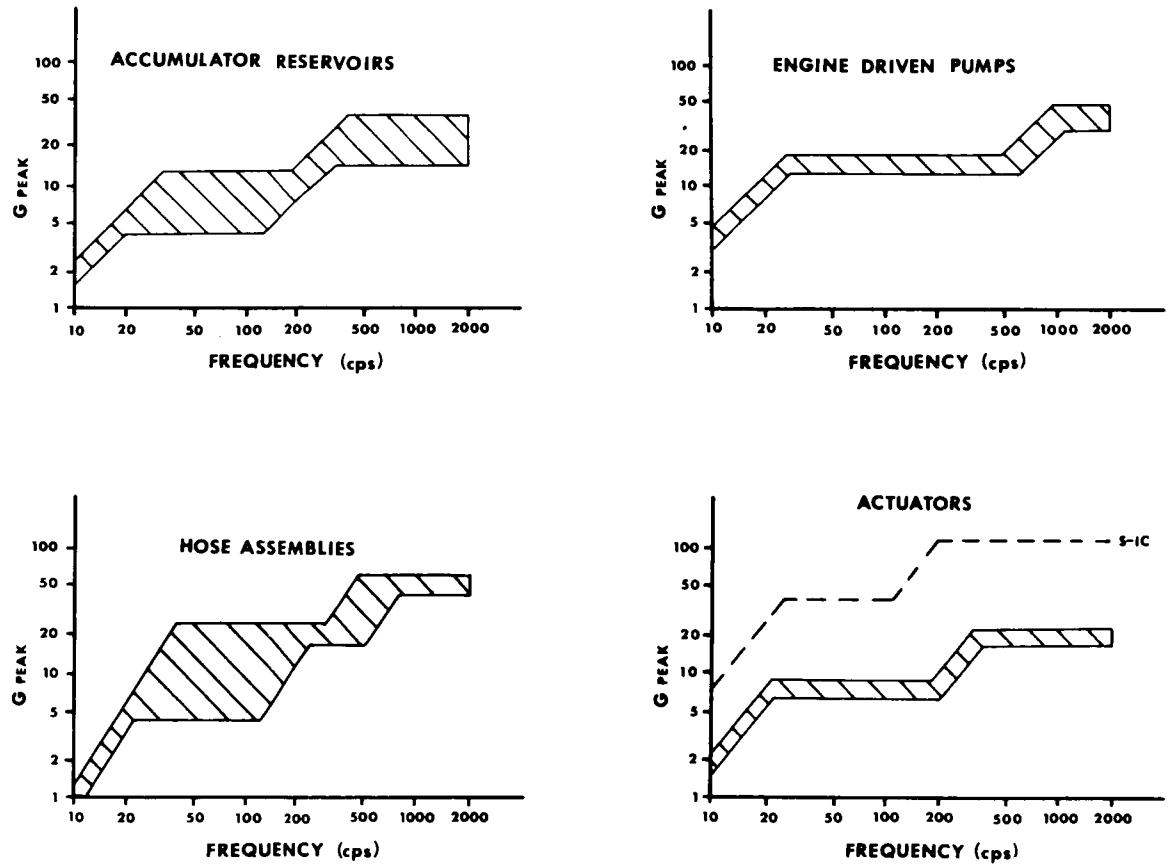



Figure 15. Saturn hydraulic components vibration levels

Programs conducted and sponsored to improve contamination controls for fluid power systems are as follows:

1. Evaluation of various types of filters and media.

2. Hydraulic component tests to determine contaminant generation and tolerance level.

3. Evaluating and testing automatic particle counters, contamination monitors, and silting index devices.

A number of design improvements of the hardware enabled additional gains to be made. Seven of these are as follows:

1. By employing fully supported filters, cantilevered or resonating ends were avoided. Figure 16 shows a filter element which can be properly supported. The release of MIL-F-8815 also permitted a better component to be specified. ARP 599 became a successful method for cleaning and monitoring filter cleanliness.

2. Employment of a standard fluid sampling valve for all Saturn systems is a requirement. Work done by several research organizations showed this valve to be adequate for the taking of representative samples.

3. A double sealed, vented accumulator has become standard for these systems, since it prevents nitrogen from getting by the dynamic piston seal into the oil. This also made the use of a ground servicer unnecessary.

4. Monitors for filters, such as differential pressure transducers and indicators, have been employed for key filter elements.

5. A precision MC fitting is used extensively on all the hydraulic systems. Since the fitting is primarily employed to minimize leakage, a secondary benefit to be derived from its use is the minimal contamination generation rate that can be expected during assembly and use. The special care and handling given this fitting contributes to the overall contamination control program.

6. Aerospace pumps and servo-actuators have been improved on the component level. "Contaminant tolerant" pumps and valves are available. Materials now being used in pumps have less tendency to erode or wear. Harder surfaces are employed in critical areas. Filters are included in pump modules.

7. Some general improvements have been made in servo valves that help to combat the contamination problem: These include fluid isolation of the torque motor; elimination of a single stage sintered bronze filters in favor of a well supported mesh type element; and mechanical feedback between the spool and valve first stage. The higher electrical power input of the mechanical feedback actuator used in Saturn V resulted in improved contamination tolerance and higher spool driving forces.

Valve reliability for the S-IVB is carried further by use of multiple components. It is estimated that this redundancy improves the servo valve reliability by 20 fold. Some of this gain can be applied to contamination control, since many of the critical elements, such as pistons, orifices and nozzles are made redundant. Since the S-IVB Stage is not multi-engine, this concept appears to have a number of obvious advantages.

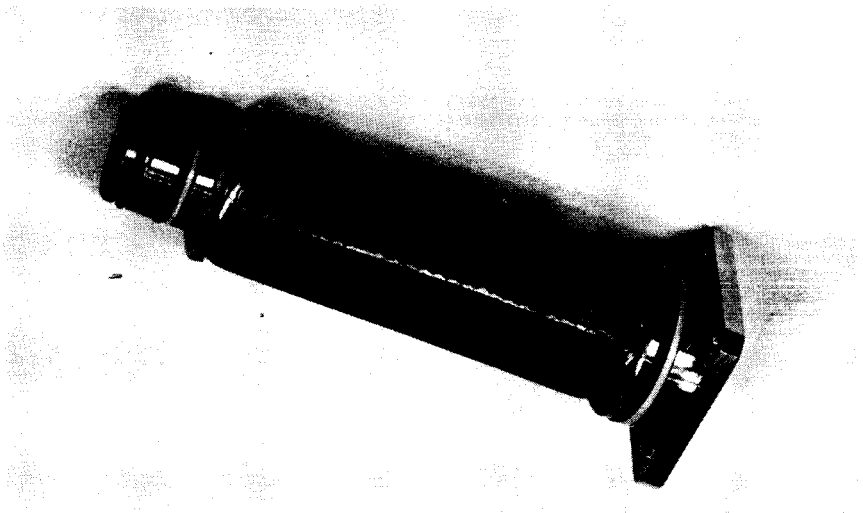


Figure 16. Well supported filter element

Possibly the best example of implementing a comprehensive contamination control program is illustrated by the S-II engine actuation system built by North American. The build-up and installation sequence is shown in Figure 17. Components are thoroughly flushed with clean hydraulic oil in a dust-controlled area. During the flushing procedure, the components are vibrated on shake tables to loosen contaminants. All the major components are actuated to flush fluid through them. All components, lines, fittings are assembled into the system before stage installation.

The completed system is then flushed in two phases, first, with the actuator prefiltration valves open, and then with them closed. After the first phase, fluid samples are taken upstream of the actuators to verify the contamination level and after the second phase, samples are taken downstream of the actuators. The entire system is moved out of the clean room on its installation fixture and installed on the stage. No fluid lines are disturbed in the process and the system remains in a sealed condition through checkout, static firing, and launch, unless a major hydraulic component needs to be replaced.

Figure 18 shows the specification system level which is being met on the S-IB hydraulic system. It should be noted that the S-II and S-IVB systems are also meeting this level.

One of the major problems relating to the S-1C fluid power system was the unknown factor involved in using the propulsion engine propellant (214,000 gallons) as the operating fluid. Contamination control became an important aspect of this problem and also became a formidable argument against using a direct propellant bleed system. Here again, existing specifications were employed, with important relaxations because of the decreased sensitivity of the servo-actuators and the absence of internal generators, such as pumps or accumulators. System filters must be able to maintain a safe contamination level in a single pass while the previously discussed systems could do this during multiple cycles. Careful contol of fuel tank cleaning and fluid furnished to the vehicle enabled the design objectives to be met. The ground system fluid was controlled to a more stringent level so that lengthy check-outs and cold gimbaling could be achieved without impairing or degrading the flight system.

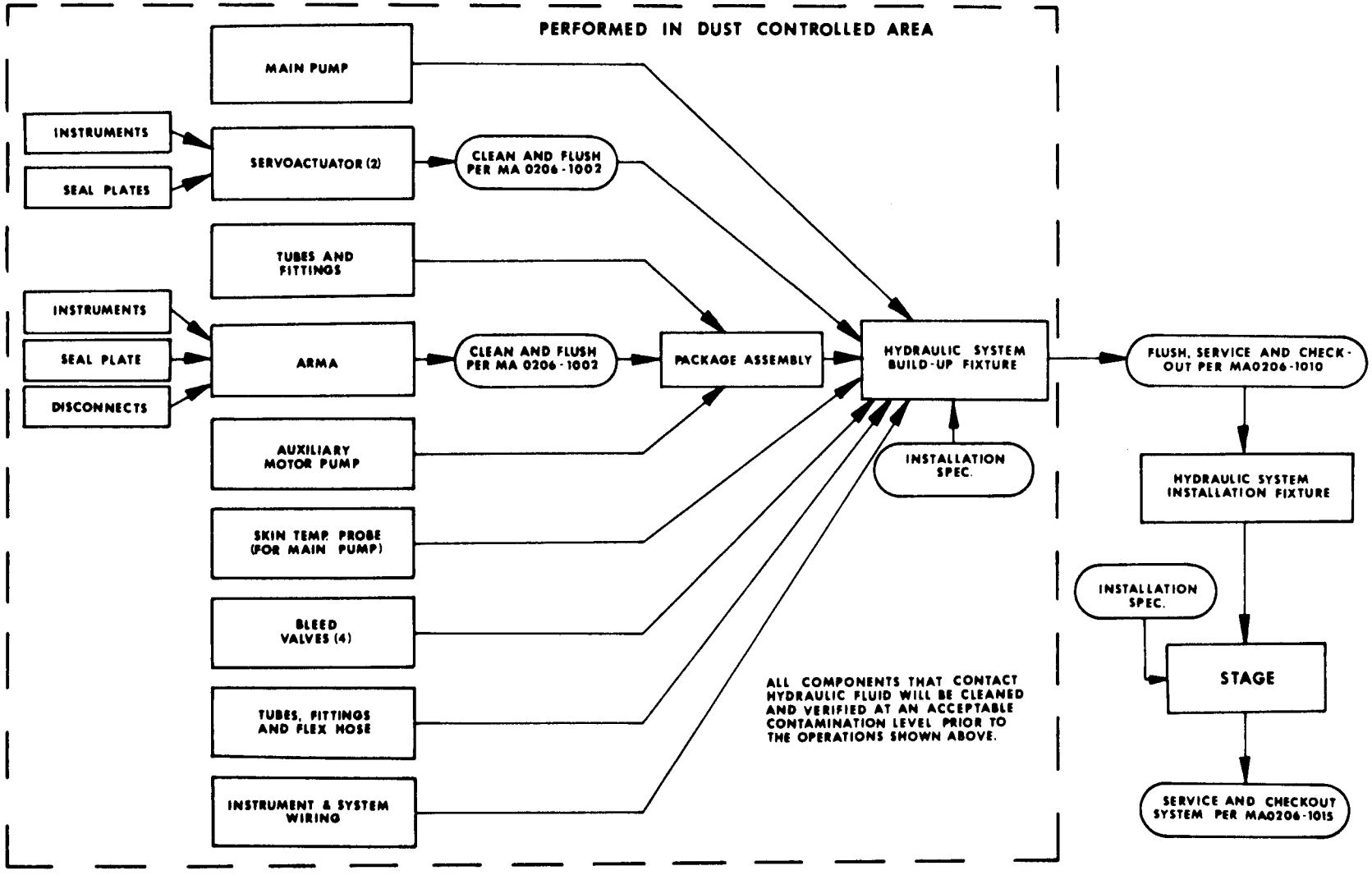



Figure 17. Engine actuation system build-up and installation sequence

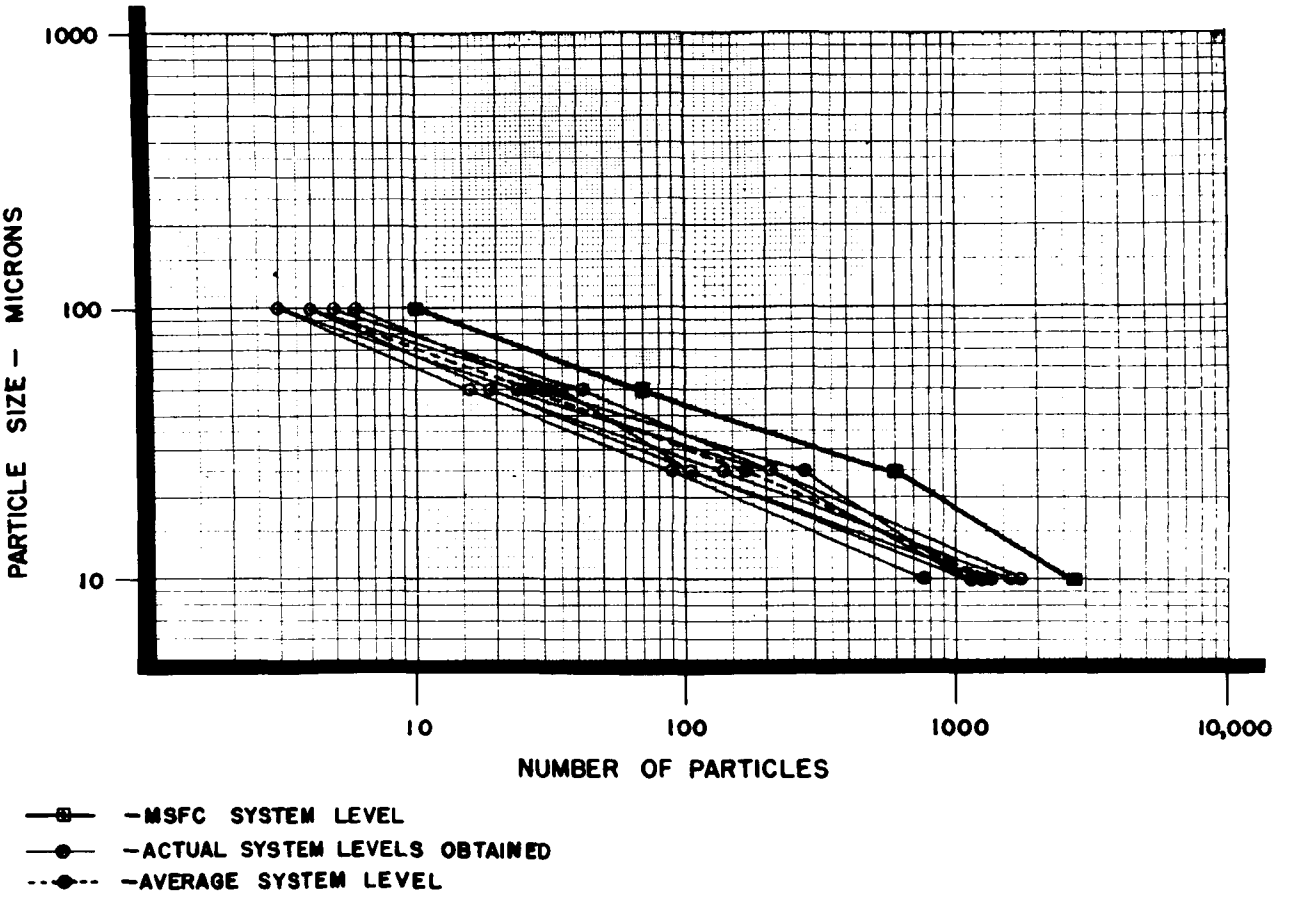



Figure 18. S-I hydraulic system contamination levels

A back-up concept considered for the S-1C system was the "wash filter" shown in Figure 19. Here the filtered flow was tapped-off for the actuation system while the main flow washed the contaminants downstream into the turbopump. With this filter, very highly contaminated fluid from the tanks could be converted to clean fluid for the engine servo-actuators. Tests were conducted to verify this concept, but conventional filtration as described above proved adequate to meet all performance requirements. Figure 20 shows the established system level and the levels obtained during the static test program of this vehicle.

## Future Considerations

Everyone envisions an automatic method for determining the contamination status of a hydraulic system. Limited go, no-go monitors do exist but cannot be universally employed at this time. Since there are always instances where systems will be broken into, automatic system monitoring may prove frustrating.

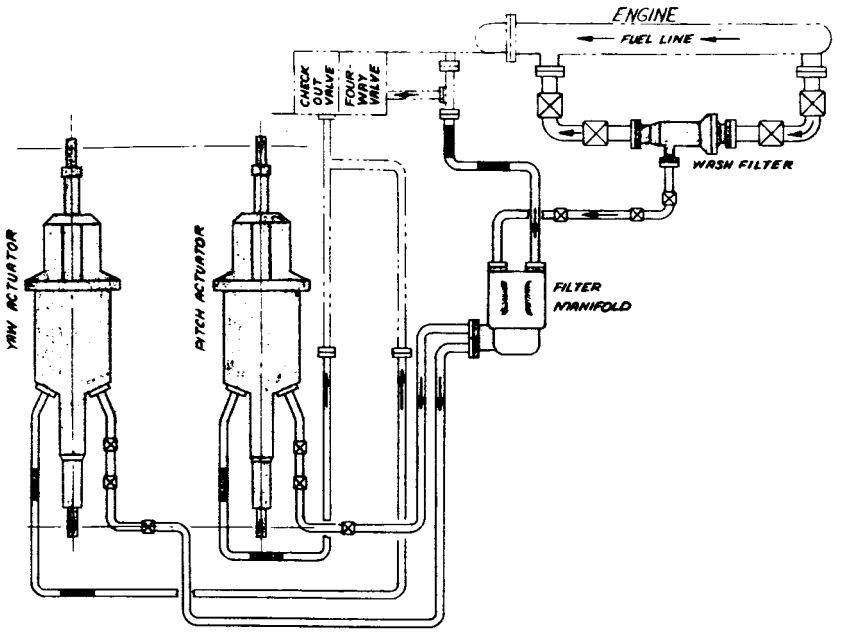



Figure 19. F-1 engine gimbal system for Saturn 1C

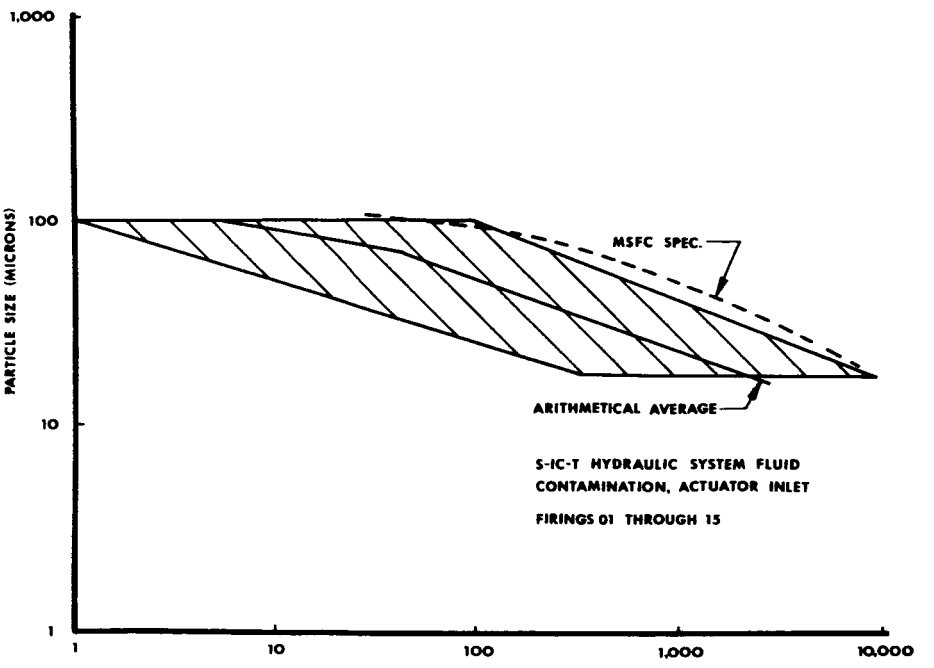



Figure 20. Accumulative number of particles of indicated size and larger per 100 ml

Automatic particle counters have usefulness as laboratory tools where highly trained operators are present. Utilization as production controls or as close coupled system monitors has not been established with any degree of universality. Also the gravimetric ingredient of the system level cannot be determined. Absolute correlations between ARP 598 counting and automatic counters is difficult to attain and maintain. Employment of particle counters in the field where the technical competence and attendant care is degraded in favor of speed will probably result in unreliable results.

Combining both gravimetric and particle count methods into one universal standard may prove to be a major milestone in this field. If a meter or contamination sensitive device can measure the combined contamination effect in a system, then many of the stringent controls presently imposed can be relaxed.

## Conclusions

Contamination no longer evokes the fears it once did, mainly because the effects and controls are better understood. Still, any system abnormalities or complete failures are often blamed on contamination, especially if inadequate data or intermittent operation is obtained. The many successful Saturn flights have proven the reliability of "closed loop," hydraulic systems.

There is no magical solution to contamination control, just carrying out the available technology and procedures in a straightforward, methodical and painstaking manner. There is no short cut to this solution for if the controls are violated often enough serious consequences will result.

A more automatic method of determining the status of a system is required. One that is quick to perform, reproducible and considers the many conditions interacting within a system.

## Question and Answer Period

MARSH: Mr. Vickers, you indicated that the automatic NVR nephelometer is more accurate and faster than the manual gravimetric methods. Yet you concluded that this has not replaced the manual technique as the standard method. Is the reason primarily because of maintenance and reliability problems with the present instrumentation?

VICKERS: Yes. Equipment maintenance problems are the barrier to the general use of the method at this time. We have had very few mechanical problems. Most of the problems are electrical, and it is felt that in the near future these problems will be resolved.

OGLE: What lamp do you use and what life do you expect for this lamp?

JACKSON: The lamp is a microscope, 12-filament, 6-bulb type. The life of the lamp has not been determined, but Mr. Vickers reports that so far there has not been any failures. A data sheet on "Lamp

Selection and Modification" will be sent to interested persons upon request to me at IIT Research Institute.

VICKERS: Although there have been no problems with the lamp to date, as pointed out in the presentation, there is a slight deterioration from use, and compensation for this is made by calibration. But we have never lost the lamp so that we had to replace it.

KILLION: What volume of solvent is used?

JACKSON: The pump rate is 15 cc's per minute. After the 15 cc's, about 14 cc's are passed to waste back into the drain to make certain there is no concentration of the contaminated solvent in a nebulizing cup.

KILLION: How can the nephelometer be used in a production area?

JACKSON: There are three modes of operation. The first is to measure the absolute contamination level of a solvent, and this is done by using either nebulizing cup A or B. The second method is sequencing, and this goes from cup A, then to cup B, back to cup A and back to cup B. The third method is the differential mode, wherein one looks at cup A, stores the contamination level, looks at cup B and stores that signal, subtracts from two and prints out the difference. The batch system, as Mr. Vickers pointed out, requires about 30 minutes to complete. With this system, once the equipment is coupled permanently to an in-line system, readings should be obtainable every minute. With the first method, using cup A or cup B, one can look at one solvent cleaning process or at the outlet from the still; with the second, sequencing, one can look at two cleaning processes or two stills; and with the third, differential, one can look at the inlet and outlet to process.

CLEMENT: How might the sensitivity of the equipment be improved in the low contamination concentration region, that is, less than 10 ppm?

JACKSON: One way to improve resolution would be to increase the signal to noise ratio. There is a level of about 30 millivolts for noncontaminated solvent; the reason is the incomplete darkness of the field on the forward scattering, along with some rate of scattering and general splurge as the aerosol is passed through the system. If the photometer were modified to reduce the amount of forward scattered light, then the signal could be further amplified, and this would give better resolution sensitivity at the bottom end.

HILDEBRANDT: On the nephelometer, when you manifold sample A and B into a common route through the photometer, do you preflash the common route to keep sample A or B from contaminating the manifold?

JACKSON: The pipe work is not appreciably contaminated by 2-3 micron aerosol since there is little impaction of the aerosol on the sides of the tubes. We can sample from cup A, then from cup B, and back to cup A and get very little, if any, cross contamination from one to the other. The sequencing method allows sample A aerosol to go through and get measured, and, then, after sample A is switched off, sample B is run through. So there is some purging of the lines. However, if one wants to use dissimilar aerosols, one through side A and one through side B, it may be wise to modify the timing sequence and positive flushing air through the system; this is not difficult to do.

WEERSING: Have you experienced difficulty in maintaining the cleanliness of the plumbing, particularly solenoid valves?

JACKSON: No difficulty has been experienced in cleaning the plumbing once the system has been cleaned out. The solenoid valves are Teflon seated, and they contain no grease.

GAYLE: What amplifier system is used on the nephelometer? The result of the characteristics on the calibration curves will depend on the type of amplifier used.

JACKSON: The nephelometer system has a linear amplifier. Therefore, system readout is directly related to photomultiplier tube output.

GAYLE: All of us who are involved in cleaning operations have an interest in the nephelometer, primarily because manual methods are not sensitive and are time consuming. Last week, a nephelometer was delivered to our laboratory for checkout. Were we premature in obtaining it, or is the state of the art far enough along so that this instrument can be used routinely 24 hours per day, 7 days per week? We are not interested in doing any R&D.

JACKSON: The concept of this device was identified some time ago, and it was designed for continuous monitoring. Following a breadboard model and first prototype, the latest development model was delivered to NASA this year. IIT is not a manufacturer, but every effort was made in the design state to make this an instrument which could be used routinely on a production line. The system still contains some mechanical and electronic difficulties. A manufacturer, if he does not change the concept radically, should be able to build in reliability quite easily.

VICKERS: One of the big problems in the instrument is the workmanship on soldering. Mr. Jackson is accurate in his statement that if an equipment manufacturer made this instrument it would work much better. Since it is a new instrument and automatic, it has to have some "bugs" in it, but these should be ironed out shortly. Also, it cannot be compared to the instrument Mr. Oswalt described because it was built for a different purpose.

JACKSON: The systems we are dealing with are highly aerosolized, highly flammable liquids. They may be spontaneously combustible in air, or, certainly, a spark could cause rapid reaction. We suggest that on the nephelometer system that nitrogen or some other noncombustible gas be used.

GAYLE: Is it part of the procedure that inert gas be used?

VICKERS: We started using an air pump as part of the system, but we changed during installation, and we have started using filtered nitrogen.

GAYLE: Maybe by the time we have another symposium, all of us will have this instrument in our laboratories. Even if this particular device is not an in-line instrument, it will be valuable simply as a replacement for the manual method of performing NVR on laboratory samples.

LIEBERMAN: Does plastic tubing cause problems with stopcock lubricant or plasticizer?

OSWALT: Plastic tubing could cause problems, but the stopcocks are degreased to remove all the lubricant. Teflon tubing is used to prevent an attack from a solvent such as PBC. If it were not for the water, polyvinyl alcohol could be used.

HILDEBRANDT: In what capacity does Sandia use chloroform?

OSWALT: Chloroform has been used in cleaning processes.

HILDEBRANDT: For in-line sampling when the fixture is opened for microscope reading, how is contamination from fallout prevented?

PICCONE: The fixture is not taken completely apart, but is loosened just enough to permit rotation. When the sample flow is finished, the springs produce a slight separation of the halves. The Belleville spring still bears on the solid portion of the membranes support and the circumferential clamp is only loosened. This design prevents any contamination from falling out.

BALLARD: What types of component or system failures have been traced to fluid contaminants?

PICCONE: Assuming that the question refers to residual contaminants from cleaning operations as well as contaminants introduced in the operational fluid, the following are typical of failures that have occurred:

a. Moisture in gases caused regulators to freeze up.

b. Iron nitrate dissolved in nitrogen tetroxide precipitated out due to temperature drop and plugged orifices.

c. Tiny glass beads used in various cleaning and testing operations caused failures of pulsating pressure regulators.

d. Burst discs and filters were corroded by residues from chlorinated solvents.

e. Pressure switches have been plugged by corrosion products resulting from the combined action of methanol, water, and hydrazine-type fuels on metal.

BALLARD: Are particulates a more serious contaminant than nonvolatile residue for your applications?

PICCONE: Yes, since we are dealing with fluids that are relatively nonreactive or are good solvents for contaminants. However, for fluids such as oxygen or fluorine, nonvolatile residues are probably more serious.

CLEMENT: What specification requirements would you place on an ideal contamination sensor? What would be required technologically to develop one?

PICCONE: Answers to these questions are not simple - nor can they be brief. The best answers to these questions can be found in the final report to the study sponsored by NASA-Kennedy under Contract NAS 10-2693.

Chapter V of this report presents detailed information on the requirements for sensors to be used in all the fluids discussed in the paper.

GAYLE: In general, we found that when we get a system or solution clean enough to pass the particulate contamination level it will also pass the NVR. Do you feel that we are going to get into a situation where sampling and analytical methods are automated to the extent indicated by your study?

PICCONE: It is doubtful that we will ever get anywhere close to what was proposed. One reason that this study has not been pushed further is that it is an expensive operation. Everyone has been getting along fairly well by the batch methods of sampling and these seem to have done a good job. While the automated methods offer a lot of advantages, there is a cost factor to consider. Automated monitoring of hydraulic systems may come into being some day because it has been demonstrated that these counters work well with hydraulic systems as long as their limitations are understood.

GAYLE: You went to a lot of trouble to develop essentially an in-line microscopic particle counter and then did not build it. It is hard to imagine getting that far along and then somehow not doing something to build it and check it out. The problem, no doubt, is funding. Can you give an idea of the cost and do you have any expectation of building the counter in the future?

PICCONE: The cost figures are on the order of $10,000-$20,000 for the complete development--design, building, testing, and several redesigns. We have proposed to the Air Force that we be allowed to build one for monitoring hydraulic systems on the TITAN since the Air Force has tightened the criteria for hydraulic systems on the man-orbiting vehicle program. We do not think we can meet the criteria unless we use a closed system. We are waiting for an answer. On the other hand, the Air Force may ease up on the criteria instead of allocating funds for the counter.

JACKSON: Mr. Smith, how did you initially clean the inside of the teflon, copper, and stainless steel tubes? Is $\Delta R$ a function of time?

SMITH, V. I.: We considered going through a complex cleaning procedure on the tubing, but we did not do this because we felt that it was not too significant. If we had virgin copper, which is essentially unavailable, we may have dissolved much more copper than we did. We did not get enough to detect, so we did not do any cleaning other than to run the still and solvent through the tubes for several hours before we made any measurements. The time seemed to have no effect. The temperature was also changed slightly. The $\Delta R$ did not change appreciably with temperature.

Q: Is this conductivity measurement technique good only for traces of impurity? In other words, you are not worried about molecules.

SMITH, V. I.: This technique is good for any level, although we have not studied contaminants to the extent that we would like to do. The electronics industry is more interested in a much higher grade of contamination-free systems than the space industry. The electronics industry is interested in molecules and particles. The contamination in some circuits is down to a particle of 0.5-1 micron in width. To the electronics industry, a 10-micron particle is a gross particle,

whereas in some space applications, it does not matter how many particles there are between 1-10 microns.

GAYLE: Mr. Smith, your resistive techniques suggest the possibility that they might be exploited to other areas of contamination. A lot of us would be interested if this approach were pursued.

COMMENT: One in-line filter has been constructed with flow into a very thin filter holder. It is very simple and very small. It is about 1-inch thick and is as large in diameter as necessary to hold the filter.

MCDONALD: That filter was developed by a petroleum company for looking at hydraulic oils and it is probably not suitable for high pressure lines and cryogenic fluids.

SMITH, S. L.: On checking the resistivity of the solution, do you know whether the contaminants go into solution, or does the change in specific gravity cause them to go to the bottom or top and explain the difference in the reading?

SMITH, V. I.: There was nowhere near enough contaminant to separate. For example, water added to freon is relatively invisible; in freon, it is soluble to about 300ths of one percent. The mechanism of resistance measurement is a facet of an electron from one side to the other. This has to be done through the liquid by some mechanism. We do not know what the mechanism is. We do know that it is changed by impurities, whether they be liquid or solid. It does not make any difference to the user whether the cause is particulate matter or dissolved water vapor; he does not want it there, especially if he can obtain the solvent pure without too much difficulty.

SESSION II

RADIATION MONITORING

Presentation

METHODS AND INSTRUMENTATION FOR MONITORING RADIOACTIVE CONTAMINATION -- F. L. Fey

Session Chairman

H. D. SIVINSKI
Sandia Corporation
Albuquerque, New Mexico

SESSION II

RADIATION MONITORING

# METHODS AND INSTRUMENTATION FOR MONITORING RADIOACTIVE CONTAMINATION

by

FREDERIC L. FEY, JR.

Ph.D. Candidate
University of Minnesota
Minneapolis, Minnesota

ABSTRACT

Monitoring radioactive contamination is unique since it is done by detecting the particle it emits. The principles involved in detecting these particles are summarized and three types of instruments are discussed: gas ionization, scintillation, and semiconductor detectors. Finally, use of these instruments and methods of radioactive contamination monitoring are discussed.

## Fundamental Principles of Radiation Detection

Before describing the instruments available for detecting radioactive contamination, it would be worthwhile to discuss the fundamental principles involved and why it is desirable to detect radioactive contamination. Radioactive contamination is composed of a quantity of unstable atoms, generally referred to as radioisotopes, which decay to a stable isotope by the emission of one or more of the ionizing radiations called alpha, beta, or gamma. It is desirable to keep radioactive contamination to as low a level as possible for the following reasons: (1) if the contamination is present in sufficiently large quantities it could cause high external exposures to personnel or even cause electronic instruments in the vicinity to malfunction; (2) if the contamination is loose, an additional hazard is present since there is the possibility of the contamination getting into the human body and causing biological damage or spreading into areas where a low radiation background is desirable, such as radioisotope counting rooms.

Radioactive contamination can be a hazard even though the actual amount of material present is very small. In microbial contamination one is dealing with cells, but in radioactive contamination, one is dealing with a relatively small number of atoms. These atoms emit an ionizing particle which does the damage. Ionization is the removal of an electron from an atom, thus leaving a negatively charged electron and a positively charged ion. Radioactive contamination is measured by detecting this ionizing radiation.

Three general principles are used in detecting radioactive contamination: (1) gas ionization, (2) scintillation, and (3) semiconductor detectors. The gas ionization method makes use of a chamber with a center electrode to which an electrical voltage is applied. Any ion-electron pairs generated within this cavity by incident radiation will be collected, thereby causing a current to flow in an associated circuit which is proportional to the radiation passing through the chamber. When a chamber voltage is used which is adequate to collect all of the ion pairs released by the radiation, the chamber is being operated in what is called the ionization region. A sufficient additional increase in chamber voltage can result in the electrons being accelerated enough that when they collide with molecules of gas in the chamber more electrons are released and collected. Thus, various degrees of multiplication or amplification can be obtained, depending on the voltage used on the collecting electrode and the geometry of the chamber. This increase in current allows each individual interaction to be detected as a very short pulse. The chamber is now being operated in what is called the proportional region, in which each interaction produces a larger signal than when the chamber is operated in the ionization region, but which is still proportional in size to the initial ionizing event. When the voltage is increased sufficiently each interaction causes an electrical discharge, like a spark, to jump through the chamber, resulting in a very large signal. The chamber is now operating in the Geiger-Mueller region, and the same size pulse is produced regardless of whether the ions for a large number.

The second type of detector, the scintillator, makes use of the fluorescence caused in certain materials by ionizing events. The light is measured by a photo-multiplier tube which gives an electron pulse proportional to the light generated in each event. The advantages of this type detector are as follows: (1) high sensitivity, (2) output proportional to energy deposited, (3) phosphors sensitive to particular radiations, and (4) liquid or solid scintillation material.

The third type, which is just now coming into use, is the semiconductor detector. This detector uses the same principle as the gas ionization chamber; however, instead of ions being collected from a gas, they are collected from the solid material of a semiconductor. This detector has the advantage of a very high energy resolution, but its sensitive area is yet too small for certain practical uses. Although other detectors, such as film emulsion and thermoluminescent dosimetry, are available for special application, the three methods mentioned are used greater than 95 percent of the time for radioactive contamination detection.

In brief, this is what can be done with the signal produced by these detectors. Most instruments in use not only detect the contaminant but give at least a relative indication of how much is present. If the contamination level is low, it is sometimes convenient to count each individual event with a scaler for a certain length of time. This technique is usually employed in a laboratory where low level samples are brought in for accurate counting. However, most portable survey instruments use a count-rate circuit in which the signal is electronically averaged and continuously displayed on a meter in terms of events per unit of time. In many instances it is convenient to connect earphones to the instrument through which the surveyor may hear the individual amplified pulses and note the reading on the meter only when significant levels are reached, as indicated by an increasingly rapid average pulse rate.

## Instruments Available for Contamination Detection

The principles discussed above may be used in a variety of instruments, depending on the contamination to be measured. Most contaminants can be classified either as alpha or beta emitters, with both usually emitting gamma rays also. Because gamma-sensitive instruments are subject to high background, it is desirable to use an instrument sensitive only to alpha or beta radiation. A few situations will be mentioned later where it is necessary to use gamma-sensitive instruments.

The most popular alpha detectors have been gas proportional probes connected to portable count-rate meters. Since these units are operated in the proportional region, a pulse caused by an alpha particle will be much larger than one caused by a beta or gamma ray. An electronic discriminator circuit allows only the largest pulses to be counted. In a dry climate where no change in altitude is anticipated, it is possible to use air at atmospheric pressure as the proportional gas. However, when either change in altitude or high humidity is involved, it is more reliable to use a special gas, such as propane, which is passed through the probe at a low flow rate. The detector chambers are typically 20 cm long by 5 cm wide by 2 cm thick. One of the large-area walls is a very thin sheet of plastic with a conductive coating through which the alpha particles may enter the chamber and be counted.

An alpha detector which is not affected by atmospheric conditions is the scintillator. These units are composed of an alpha-sensitive scintillation material with a thin, opaque covering to keep out light but admit alpha particles. The light produced by each alpha interaction is measured by a photomultiplier tube and the resulting electrical pulse rate is indicated on a count-rate meter. These instruments do the job very well but are perhaps not quite so rugged and are a little more expensive than the proportional-chamber detectors.

The instrument most commonly used for detection of beta-active contamination is the Geiger-Mueller tube. This steel-walled chamber operating in the Geiger-Mueller region, provides high sensitivity to to every type of ionizing interaction. The steel wall is normally made thick enough to keep out alpha particles but thin enough to admit beta particles. Since this instrument is also gamma-ray sensitive, it is subject to background count due to natural radioactivity and other sources of radiation in the area. The Geiger-Mueller probe is typically an 8-cm-long tube about 2 cm in diameter. The tube is contained in a metal housing with a rotating portion which allows one side of the tube to be either opened to detect beta particles or closed to detect only gamma rays.

A beta-sensitive scintillation detector could be used in the same way the alpha scintillation detector is used; however, the Geiger-Mueller detector has done the job so well that there has been little call for a better instrument.

There are a few radioisotopes which emit gamma rays but no alpha or beta particles. Also, under certain conditions alpha contamination is very difficult to detect with an alpha-sensitive instrument because the alpha particles may be absorbed in a wet or dirty surface before

they get to the detector. In both of these cases it is necessary to rely on detection of any possible gamma rays emitted. A Geiger-Mueller tube is quite sensitive to gamma-rays and it will serve adequately in many cases. However, the use of a scintillator crystal has the added advantage of high energy resolution, and discrimination against background radiation or even selection of a certain gamma-ray energy can be provided.

Semiconductor detector characteristics offer very high energy resolution in alpha, beta, and gamma detection. When it is possible to make this type of detector larger and more efficient, it will undoubtedly become more popular.

## Methods of Contamination Detection

The instruments mentioned above can be used in different configurations, the most popular being a hand-held survey instrument. The probe is manually moved over the surface suspected of being contaminated. The detector is kept as close as possible to the surface without touching it to provide greatest sensitivity, yet avoid contamination or damage to the probe. This is especially true when alpha contamination is being monitored because an alpha particle travels a very short distance in air.

Special equipment has been designed for monitoring large, flat surface areas such as floors. These are usually a string of several Geiger-Mueller tubes or one large gas-proportional probe mounted between two wheels with a count-rate meter attached to a handle. The instrument is rolled over the surface to be monitored while the surveyor observes the pulse rate.

A similar instrument called a portal monitor is used to monitor personnel automatically for contamination as they leave potentially contaminated areas. Personnel walk through a door frame containing a series of Geiger-Mueller tubes around the periphery. If the count-rate exceeds a preset level, the unit alarms. The portal monitor could be used to monitor a large number of similar items; however, in order to detect alpha contamination, the probe would have to be kept very close to the surface being monitored.

Detecting contamination on inaccessible surfaces is not so straightforward as detecting it on accessible surfaces. An inaccessible surface may be checked by rinsing or even washing the surface thoroughly with a suitable solvent and then analyzing the effluent for radioactivity. Air samples may be taken in certain areas where there is the possibility of loose contamination. These air samples can be taken to a counting laboratory, where accurate counting techniques can be employed to determine whether there is radioactive contamination in the air. Air can be drawn through a filter, which collects the particulates. After some collection time, which might be many hours, any activity on the filter is measured and the activity related appropriately to the actual air contamination. This can be a very sensitive method because of the large volume of air that can be sampled. Continuous automatic air monitors are available which count the radioactivity on a filter as air is being drawn through it and will alarm when a preset level is reached.

In many cases it is desirable to find out whether radioactivity is loose or fixed to a surface. This can be determined by employing the rinse method described above or more simply by counting swipes. A piece of filter or tissue paper is wiped over the area to be checked, then counted with an appropriate instrument, such as a shielded, low-background counter. The various isotopes in a certain contaminant can be identified in many instances by analyzing a sample with an energy spectrometer, which helps in determining the source of and hazard associated with the contamination.

## Conclusion

This has been a general discussion of methods and instrumentation for monitoring radioactive contamination. Special monitoring jobs may require special instrumentation, but they will employ one or more of the principles discussed. Further information on any particular radioactive contamination monitoring problem can be obtained in the literature or by consulting a qualified health physicist.

## SESSION III

## SURFACE CONTAMINATION

### Presentations

| | | |
|---|---|---|
| 1. | SURFACE CONTAMINATION GENERATED BY MATERIALS OF CONSTRUCTION | -- D. M. Davis |
| 2. | CLEANING AND MONITORING ALUMINUM SURFACES BEFORE WELDING (Paper not Received) | -- Z. L. Saperstein |
| 3. | MONITORING CLEANING PROCESSES AND PROCEDURES | -- Mrs. J. Y. Ellenburg |
| 4. | AUTOMATED FINAL CLEANING, RINSING, AND DRYING STATION | -- J. P. McDonald |
| 5. | A UNIFIED CLEANING FACILITY IN A CLASS 100 CLEAN ROOM | -- J. A. Kenagy |
| 6. | EVALUATION OF AND REQUIREMENTS FOR AUTOMATED CLEANING EQUIPMENT | -- R. B. Hedrick |
| 7. | REVIEW OF SURFACE CLEANLINESS TESTS | -- C. W. Jennings |
| 8. | PRINCIPLES OF OPERATION OF THE INDIUM ADHESION TESTER USED FOR SURFACE CONTAMINATION MEASUREMENTS | -- G. L. Krieger |
| 9. | THE INDIUM ADHESION TEST APPLICATIONS | -- S. L. Smith |

Session Chairman

Q. T. USSERY
Manned Space Center
Houston, Texas

SESSION III

# SURFACE CONTAMINATION

## INTRODUCTION

by

Q. T. USSERY

Manned Space Center
Houston, Texas

One of the most difficult and sometimes perplexing aspects of contamination control is that of determining surface cleanliness. Another is the cleaning process and procedures used at arriving at some desired level of cleanliness. In this session, the speakers will present a series of papers relating to these matters.

# 1. SURFACE CONTAMINATION GENERATED BY MATERIALS OF CONSTRUCTION

by

D. M. DAVIS

Manufacturing Research Division
Sandia Corporation
Albuquerque, New Mexico

ABSTRACT

One type of contamination cannot be diminished by the use of clean rooms or prevented, to any great degree, by cleaning and process control. Many of the materials used to fabricate items for aerospace and ordnance are sources of contamination after processing. A number of these materials, the contamination they generate, and some of the means used to control them are discussed.

## A Situation

Recently a supplier was assembling inertial sensing devices for Sandia Corporation. Each device contained an electrical switch whose proper functioning was essential for operation of the device. Every effort was made to insure that these devices were kept clean during and after assembly. They were assembled in a controlled area by persons wearing protective clothing and hand coverings, and each subassembly was cleaned to remove any handling or process debris. Low-current and low-voltage tests of the completed units showed the contact resistance of the switch after actuation to be generally within specified limits. The units were then subjected to 16 to 24 hours of an elevated temperature soak and retested. Yet too large a percentage was rejected for high contact resistance after actuation. The switch was made with rotary slider contacts which moved between two spring stationary contacts to produce a rather large wiping motion. Contact pressure was more than sufficient and did not decrease during the temperature soak. Why did this switch, so carefully assembled, cleaned, and checked, fail to perform satisfactorily?

The contact material was a high gold and palladium alloy containing approximately 15 percent copper. Investigation showed that at the elevated temperature - approximately 160°F - the copper at and near the surface of the alloy was oxidizing to copper oxide, and copper near the surface was migrating to the surface and also oxidizing. As the switch was actuated, the wiping action of the stationary contacts scraped this oxide of copper and deposited it at the end of the contact travel. Every subsequent actuation deposited more of the oxide at that spot, and the contact resistance became higher. The situation now seems to be remedied. The supplier of the alloy is now heat-treating it in air,

***

This work was supported by the United States Atomic Energy Commission.

instead of in an inert atmosphere, and then pickling it to remove the copper oxide formed, thereby leaving the surface depleted of copper.

## Introduction

This is not an isolated case. Unfortunately, the problem of contamination is becoming more and more prevalent. Many different materials are used in constructing electromechanical components to high reliability standards, and many of these components will experience elevated temperatures sometime during their lives. Microminiaturization has increased the problem, since much less contamination can be tolerated. Therefore, I would like to discuss a few of the materials which can cause surface contamination describe several specific examples of problems, and then suggest some of the ways by which the contamination can be somewhat controlled.

The most emphasis will be placed on the outgassing of polymeric or plastic materials, and I will touch on some of the problems of migration and separation of lubricants, the oxidation or corrosion of metals, and the entrapment of sealing materials. Also discussed will be the choice and processing of materials as a means of control of surface contamination.

## Outgassing of Polymeric Materials (Plastics)

Probably the greatest number of problems encountered to date in electromechanical components, have involved plastic or polymeric materials. These materials have been used in ever-increasing amounts over the past few years because of their great weight advantage that their lower density and improved strength provide. A number of them with proper processing, have been quite satisfactoty. However, to obtain the desired physical characteristics, these chemically complex materials are often modified with additives and through changes in chemical composition. In the process, they are often made more susceptible to outgassing,[1] their greatest source of contamination generation.

The outgassing products of these organic materials generally come from one or both of two sources. One is the unbound or weakly bound materials in the polymer, such as plasticizers, some curing agents, catalysts, antioxidants, and other additives added by the supplier of the raw or unprocessed materials. The other is only an extension of the first, in which the user of the materials deviates in the processing of them by incorrect weighing, mixing, molding, or use of insufficient or incorrect cure cycle. Such deviation will yield contamination in the form of monomer, pre-polymer, curing agent, or catalysts, and can also cause the processed material to fail structurally.

The plasticizers used in many plastics are not usually tied up chemically in the polymer structure, but are merely interstitially held in place. These plasticizers, such as dibutyl phthalate or tricresyl phosphate, can easily bleed from the material, especially at elevated temperatures, and migrate to other surfaces. Most people have had a

[1]Outgassing refers to any weight loss which the material experiences, both through vaporization of volatile components and migration of nonvolatile components.

film of plasticizer form on their car windows as a result of outgassing of the plasticizer from the vinyl seat covering.

The catalysts used in most room temperature vulcanized silastics are true catalysts; as such, they are not bound into the material chemically and, therefore, can bleed from the material. Two of the older catalysts, tin octoate and lead octoate, readily corrode copper, silver, and lead. On one occasion a gold-plated copper piece was potted into an RTV silicone, with one face of the piece left exposed. After a period of time, a green, viscous film was found on the gold-plated surface. The gold plate was porous, and the very corrosive octoate salt used as the catalyst in the RTV silicone had bled from the silicone, migrated to the adjacent gold plated surface and attacked the copper substrate through the pores in the gold. Fortunately, other catalysts available are not corrosive; however, most are still able to migrate.

There are other problems also associated with silastic and other silicone materials. One is an apparent breakdown or depolymerization of the material at elevated temperatures, which results in a continuous weight loss. The outgassed matter condenses on adjacent surfaces. Another problem in the incomplete removal of reaction products during room temperature curing. In some systems this is acetic acid, in other systems an alcohol is the reaction product. If these products are not completely removed, they can outgas with time. A rumor is that one of the space walks was ended prematurely because outgassing from this type of material used as a sealant or gasket caused fogging of the visor of the helmet.

Polysulfide sealants and elastomers, as well as other vulcanized elastomers, can give off sulfide vapors that can react with metals, especially copper and silver base alloys.

High-temperature and pressure-molded plastics, although among the better materials, are not completely free of surface-contaminating materials. If they are not molded at exactly the right temperature and pressure, or if the mixture of the molding compound is not compounded exactly, the reaction is not complete; later, monomer, prepolymer, and peroxide catalyst can be outgassed if the material is not correctly processed. This occurs even though the mechanical properties meet requirements or specifications.

The outgassing or extraction of these materials becomes worse if the hard resin coat of the molded part is machined or otherwise removed. The exposed filler material is a good source of particulate contamination.

If electrical contacts or other functional surfaces are molded into the plastic, two other sources of surface contamination must be contended with. The first is the mold release agent. Many of these release agents are silicones which are polymerized onto the mold and onto the electrical contacts. The other source is a combination of the molding compound itself and a mold which is out of tolerance or worn. A thin film of the molding compound--sometimes too thin to be visible to the naked eye--will be formed over the contact surfaces. The only way to remove these contaminants effectively, if one becomes aware of them, is by using abrasives.

Often the supplier of the prepolymer will add a small percentage of some other material. The buyer is never informed of this addition,

and nothing appears in the sales literature. The supplier has tested his product, and no bad effects are noted in the physical properties of the cured polymer. Several years ago, such a contamination source was a molded diallyl isophthalate part. A number of the parts were being given a post-cure high-temperature vacuum bake, which is a standard procedure. When the oven was opened, a waxy material was found condensed on the cooler glass of the door. Analysis showed the material to be a fatty acid ester. By checking with the supplier of the molding compound, it was learned that he had been adding up to two percent calcium stearate to the prepolymer as a sort of built-in mold release agent just in case someone forgot to use a release agent in the mold. Unfortunately, this happens all too often with proprietary materials. In this case, Sandia was able to remove the surface contamination by readjusting the cleaning schedule. It had to be assumed that the parts would never see as high a temperature and as low a pressure in storage and use.

Certain of the epoxy formulations have proved quite satisfactory for Sandia use. However, whenever a modified system is required, great care must be used in its selection. If modification is accomplished by using materials, usually curing agents, which become a part of the polymer molecular structure, there is less of an outgassing problem. It is in the cases of interstitially added materials, (those not completely tied up in the molecular structure) that outgassing becomes a problem. This is a fairly good rule-of-thumb for all plastic materials.

The greatest problems with epoxies, otherwise, have been due primarily to incorrect processing, that is, incorrect ratio of hardener to resin, insufficient mixing, or insufficient cure time. One experience involved a simple epoxy system with a low molecular weight amine hardener and an alumina filler. The epoxy was placed into a narrow channel at the end of a cylinder to seal an endcap in place hermetically. A silicone O-ring was used as a backup seal. The epoxy was being applied with a syringe and needle and was somewhat too viscous for easy use. For thinning the mixture, an excess of the more-free-flowing hardener was added, instead of more resin. The extra hardener later came out of the cured epoxy, attacked the silicone O-ring, and together they contaminated the inside of the device. Someone who knew very little about epoxy systems made a decision--a costly one. Most of the time the trouble is caused by operator error and is harder to trace.

Anaerobic polymers, used extensively for locking screw threads, will have almost 100 percent weight loss if any surface area is left exposed to air. Since there is no way to ascertain that air is completely excluded from the material, we have tried to prohibit the use of such polymers in hermetically sealed components which contain electrical contacts or other sensitive surfaces.

## Contamination from Lubricants

Lubricating oils, because of their low surface tension, spread on a surface and form a thin lubricating layer or film of oil between moving parts. These oils also have very low vapor pressures, so that evaporation of the film formed is kept to a minimum. As long as the film stays on the surface where it is desired, it is performing a useful function. However, this same thin film can be a source of contamination if it should spread to a surface where it cannot be tolerated, such as at electrical contacts or the commutator and brushes of a motor. The problem is made even worse if the vapor pressure of the oil is too high and evaporation occurs, or if the oil contains some more volatile

components which evaporate. In a closed system, these evaporated materials will condense on other surfaces.

Greases must be considered in the same light as lubricating oils when contamination is involved, for the former are usually nothing more than lubricating oils milled with a soap, such as lithium stearate, to keep the oil from spreading. Even the best high-temperature greases bleed to some extent, spreading a very thin film of oil over adjacent surfaces. We have on many occasions detected traces of oil on the commutator of a small DC motor located as far as 1/2 inch from a miniature precision bearing containing approximately 3 milligrams of a Mil-G-23827 grease.

Solid-film lubricants, if applied too thickly or on a poorly prepared surface, will generate particulate contamination within a component. If they are not properly cured, they will also generate film-type contamination by outgassing unreacted materials as well as failing as a lubricant. Even properly applied and cured solid-film lubricants must be "run-in" to remove any particles generated by initial operation of the lubricated parts; the parts must then be cleaned well before being sealed into a unit or component.

## Reactions of Metals

By far the most prevalent source of contamination generated by metals is oxidation. Although all metals tend to oxidize, the tendency in a few is much greater than in others, especially with moisture present to form an electrolyte. In most cases the reaction products are easily discernible during a post-mortem examination, although occasionally a situation occurs similar to that with the previously mentioned precious metal alloy. The particulate matter associated with this contamination can disrupt the proper functioning of a device as debris is distributed throughout the interior; damage to the metal surface from pitting and reaction product build-up often causes failure of a surface, such as a load bearing or low coefficient of friction area, or the failure of the part as a structural member.

Galvanic corrosion, the result of an electrolytic couple between two metals of opposite or dissimilar cathodic potential, does not occur only in water pipes or conduits buried in the ground. If any complete electrical path is provided and a trace of moisture is present, corrosion can occur if no preventive measures, such as plating or other coating or treatment, are taken.

If any two metals are in intimate contact but are able to move an infinitesimal distance relative to each other, a phenomenon called fretting corrosion occurs. This produces a finely divided particulate contamination, usually brown in color, which is believed to result from the localized high temperatures generated by the friction between the two pieces. Vibration is the usual cause of fretting corrosion. A somewhat similar phenomenon occurs on noble metal electrical contacts in the presence of organic vapors or films. The product of this reaction is called frictional polymer. Platinum and palladium are more likely to undergo the reaction than are the other noble metals. Some other metals will form an organometallic complex with organic vapors if they are enclosed. Cadmium disease is an example; white, slightly curly, whiskers grow out of the cadmium surface.

## Contamination Generated in Sealing Processes

Certain sealing processes and materials will contaminate the interior of an otherwise clean and contamination-free component or device. Soldering and brazing are effective means for producing hermetic seals; however, neither can be done without the use of a flux which moves ahead of the molten metal and prepares a surface for it. In one place, the flux goes inside the unit to be sealed unless a good mechanical barrier has not been provided. The organic, soft-solder fluxes will leave an insulating organic film on interior parts and cause improper operation of some moving parts and of electrical contacts. The more active fluxes and the brazing fluxes are highly corrosive. A relay manufacturer has had to result to a solder back-up for repairing leaking weld seals. The corrosive flux used in getting inside the relay through the holes that are being filled. Once inside, the flux is subliming and corroding many of the inside metal parts. Initial testing of the relay does not always indicate the problem.

Many organic sealants, if they are prone to outgassing, can also get inside units being sealed. Some of the polysulfide and silicone-type sealants have already been discussed under outgassing of polymer materials.

## Contamination Control Methods

Since the type of surface contamination under discussion is a property or product of the materials used, any attempt at controlling such contamination must involve the selection and subsequent processing of the materials. There are several rules-of-thumb which will allow choice of a satisfactory material in a great percentage of cases. In all choices, however, cost must be one of the last considerations.

When choosing a plastic material, including adhesives, try to find one which has not been modified heavily with unreacting additives. This will usually eliminate many of the thermoplastic materials, such as poluvinyl chloride, which is heavily plasticized. Also, pick one which is capable of withstanding temperatures above any expected-use temperature--usually, the higher the capability, the better. A third characteristic to look at is expected weight loss. If the loss is over 0.5 percent at an elevated temperature for a period of several days, or if the loss is continous, it is safer to look elsewhere. A fourth factor to consider is the material's ability to withstand some stringent cleaning during subsequent assembly and processing. Usually, a material that will meet any one requirement will meet most of the other three. The final decision on a material usually should not be based upon the manufacturer's sales literature. Weight loss data of the kind necessary, although not always available, are fairly easy to obtain with a minimum of equipment.

Below are several weight-loss-at-elevated-temperature versus time curves which were obtained about 8 years ago by Berry, Jones, and Fjelseth.[2] All temperatures are 165°F unless otherwise noted. Most of

[2]Berry, Jones, and Fjelseth, Contact Resistance and the Effects of Materials and Process Variables on Contact Resistance and Contact Reliability in Switching Devices, SCTM 73A-60 (16), Sandia Corporation.

the materials are still in wide use today. Some new, higher-temperature materials have been developed since these data were accumulated.

Figure 1 shows the weight loss of certain phenolic materials. All show either an initial or continuous loss greater than 0.5 percent. The subsequent weight gain shown by curves (B) and (C) is due to oxidation.

All but two of the epoxy formulations shown in Figure 2 appear to stabilize after an initial weight loss, and thus are acceptable from a weight loss standpoint. At higher temperatures, the polysulfide-modified epoxy might give a problem, especially one of the corrosive sulfide nature. Curve G shows a one-component, anhydrive cured epoxy. There is a possibility of insufficient cure in this material.

Three materials shown in Figure 3 are questionable. The alkyd-glass (Curve C) is still showing weight loss after 1500 hours. The melamine-glass (Curve D) was off scale before the first weighing was made. The silicone-glass (Curve F) is showing a tendency for continuous outgassing at 220°F.

Figure 4 shows the very stable fluorinated polymers in Curves A and B. Nylon (Curve C) shows a somewhat high weight loss at 220°F, but it appears to stabilize. Moisture is usually the outgassing product with nylon. Some nylons start to oxidize at these temperatures, as did the polyethylene in Curve D.

Figure 5 shows the weight loss of several of the high-temperature molded polyesters. They all appear to stabilize after an initial outgassing period, accept that the acetal resin in Curve H shows a continuous weight loss. There have been several conflicting reports on the behavior of this material.

Figure 6 shows the very good behavior of polycarbonate. (Unfortunately, this material is quite solvent sensitive.) The silicone rubbers in Curves C through F all show large and continuous weight loss. Some of the more recent silicone rubbers are much better and capable of higher temperatures.

## Summary

Processing of a plastic must be done with great care if a stable material is to be obtained. Two- or three-component systems must be accurately weighed and thoroughly mixed if the reaction is to be near complete and the maximum physical properties achieved. The cure cycle must be followed rigidly. It is usually far better to over-cure a system than to under-cure it. If a room-temperature cure and an elevated-temperature cure are given as alternates, use the elevated temperature cure. When the weight loss curves indicate an initial weight loss followed by weight stability, we call for a post-cure, high-temperature vacuum bake on parts which can stand such treatment, to remove the more volatile by-products of the plastic materials.

It is fairly easy to select metallic materials that will produce only small amounts of contamination. There are many corrosion-resistant alloys available, and the data on them is substantial. If machinability or property (e.g., magnetic) requirements force selection of a more sensitive material, a coating can often be applied to protect the

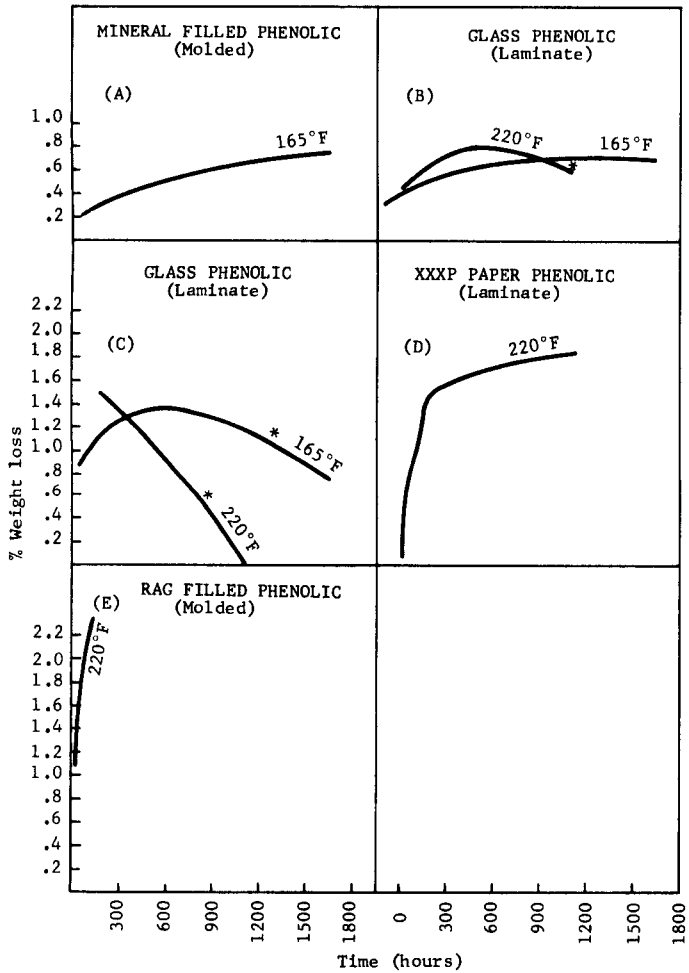



Figure 1. Weight loss of phenolic materials

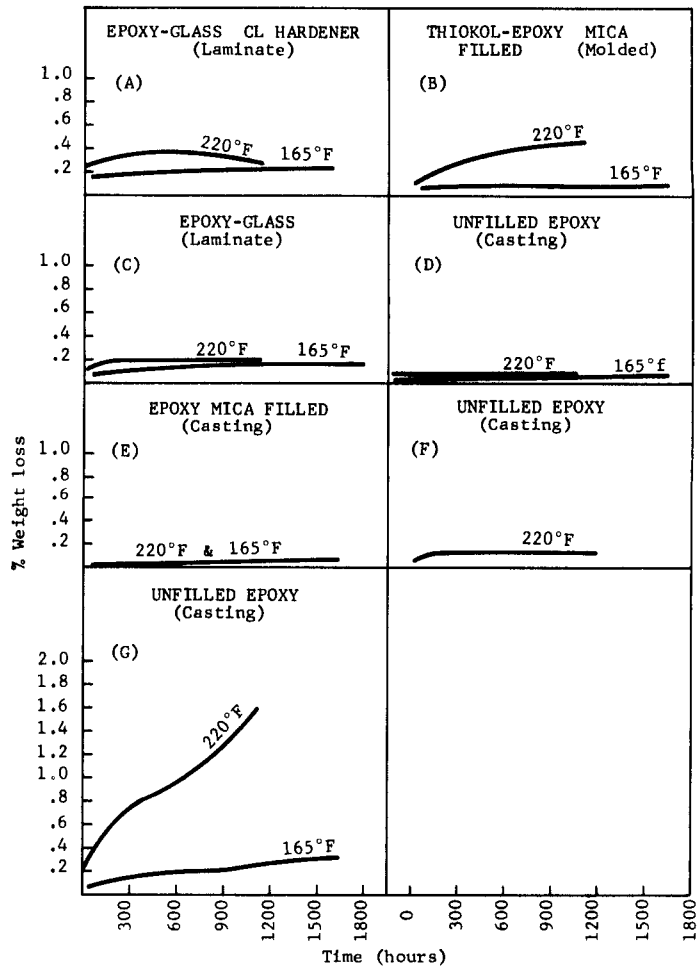



Figure 2. Weight loss of epoxy formulation

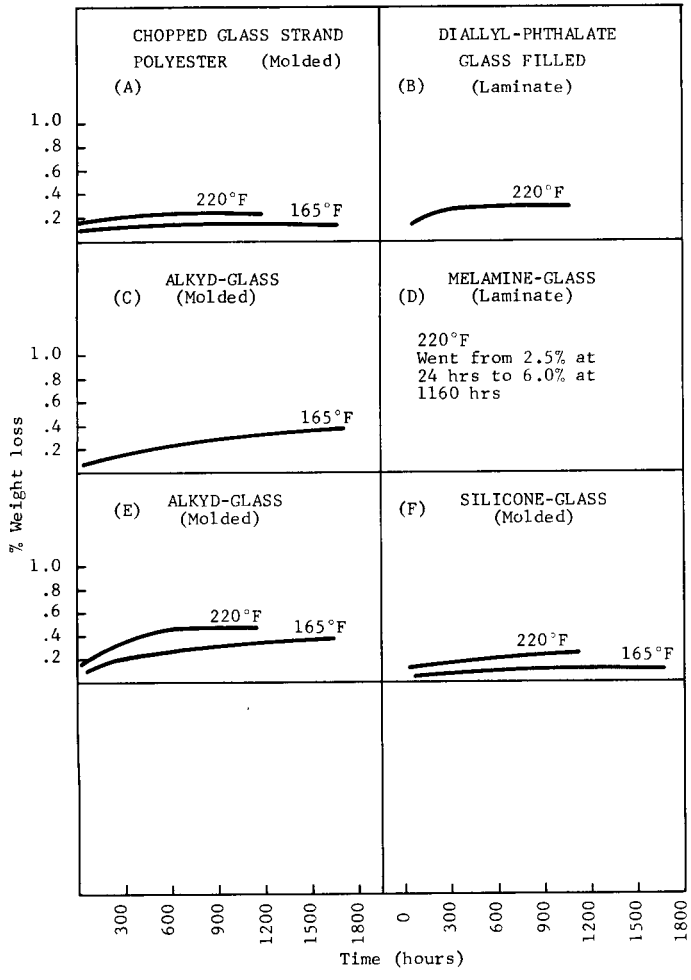



Figure 3. Weight loss of glass formulation

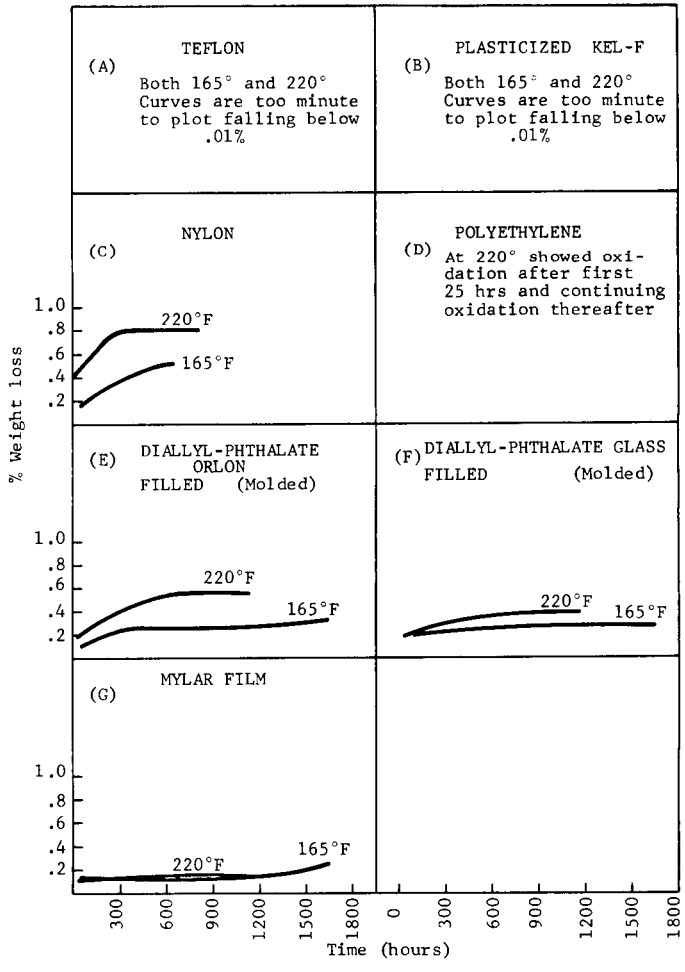



Figure 4. Weight loss of fluorinated polymers

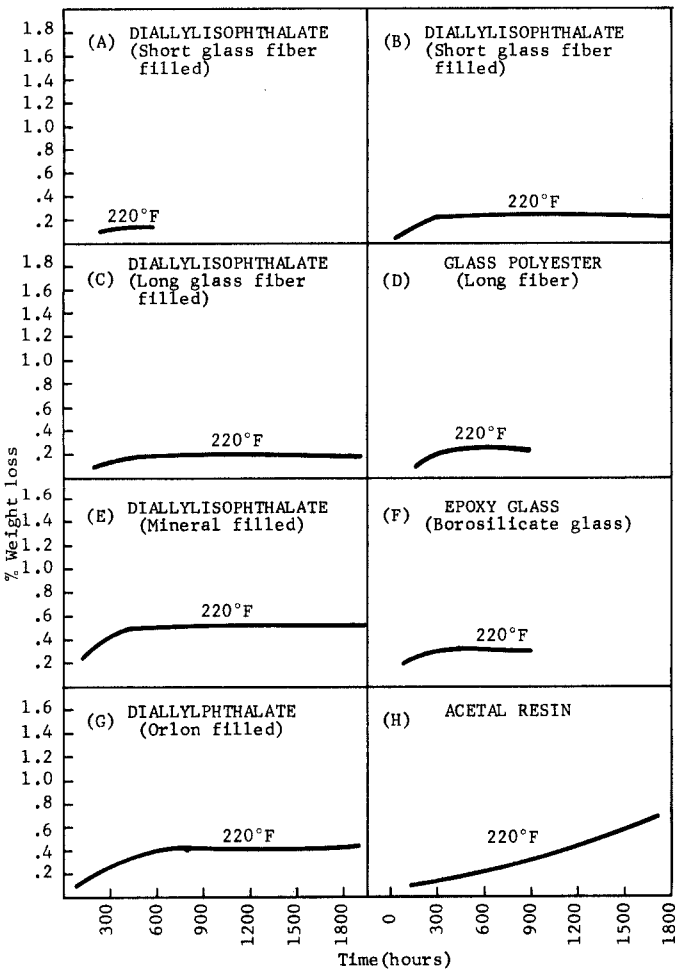



Figure 5. Weight loss of molded polyesters

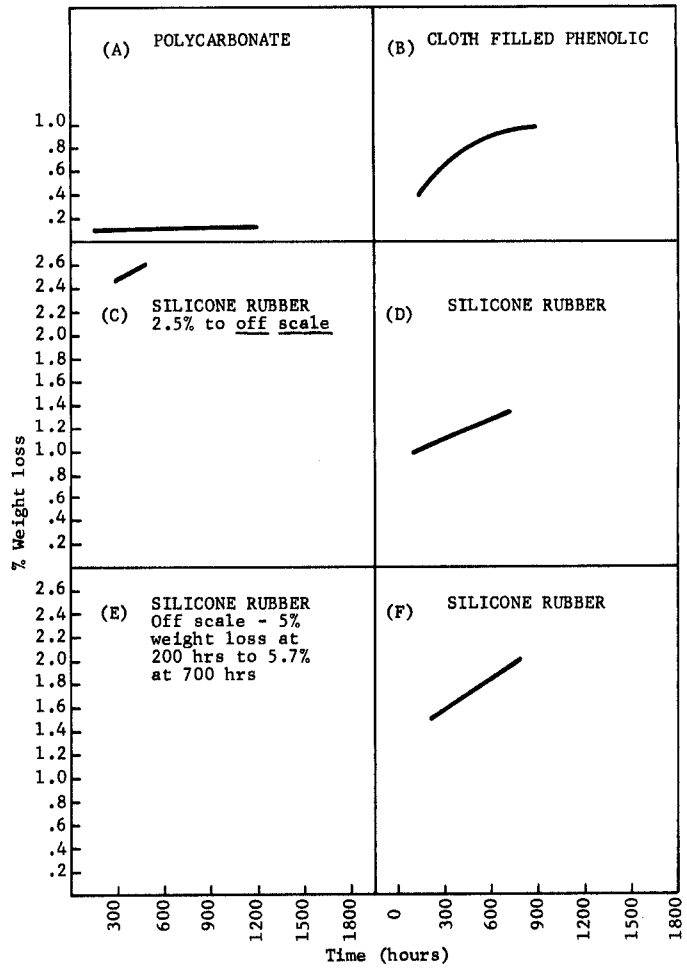



Figure 6. Weight loss of silicone rubbers

material. It is in this type of processing, however, that problems tend to occur. If a metal is passivated, often the solution is not completely rinsed from holes in the piece, and a residue of corrosive salts is left. The same problem can arise in electroplating, along with problems caused by a too thin or porous plate. The wrong coating can be used, as was previously mentioned in regards to cadmium. Coatings must be chosen and processed with the same care as all the other materials.

Lubricants should be chosen and processed in much the same manner as plastics. Choose the ones with the highest temperature capability, and then use a minimum amount. In the case of solid-film lubricants with an organic base, the thickness and cure require good control and the proper surface preparation must be provided. It is a good idea to require the same vacuum bake for lubricants as for plastic materials, since some volatiles are usually present. One word of caution, however,--do not use silicone oils and greases in areas where electrical connections are made and broken. For example, any silicone vapors present near brushes and commutators will break down in the arc in the presence of air and form silica. The brushes and commutator will not stand up very long in this environment.

Sealing should be done mechanically as often as is possible; is a preferred method. Often a simple O-ring seal, properly designed, will be sufficient for a hermetic seal. Use soldering and organic sealants only as a last resort, and then only over a tight mechanical closure. An unmodified epoxy sealant is most preferred from a contamination standpoint.

Cleaning and handling can have a strong bearing contamination generated by materials and processing. An adequate cleaning and handling schedule of parts and assemblies is necessary to insure low contamination.

Many materials are sensitive to some cleaning solutions, and the use of these solutions may make an otherwise usable material unacceptable. Processing debris must be removed if it in itself is not to cause a contamination problem. Use a cleaning sequence which will remove the particular contaminant. There is no one solvent which can do this.

Handling must be kept to a minimum. Finger oils and skin flakes can be fairly easily removed, but the corrosive salts from fingerprints can not--especially by organic solvents. Often these salts have already done their damage before they can be removed. The effect of salts is easily seen in copper alloys.

## Conclusions

A type of contamination has been discussed that cannot be controlled even by the best clean room, special handling and processing, and precision cleaning practices. It is contamination generated by the materials of construction themselves. The only control possible in this case is in the choice and subsequent processing of these materials.

Comments on these materials have been very generalized. In actual practice, after some preliminary rejection of materials by using the rules-of-thumb mentioned, each choice must be considered individually as to type and amount of contamination.

## Bibliography


1. Fjelseth, D. E., Studies of the Outgassing of Various Plastics at Elevated Temperatures, SCTM 137-58 (16).

2. Fjelseth, Davis, Jones, and Schrodder, Clean Assembly Practices Guide, SCDC-66-1118, Sandia Corporation

3. Jones, L. K., Materials and Process Variables and Their Effect on Contact Resistance, SCR-190, May 1960, Sandia Corporation.

# 2. CLEANING AND MONITORING ALUMINUM SURFACES BEFORE WELDING

by

Z. L. SAPERSTEIN

IIT Research Institute
Chicago, Illinois

(PAPER NOT RECEIVED)

# 3. MONITORING CLEANING PROCESSES AND PROCEDURES

by

MRS. JANUS Y. ELLENBURG
Hayes International Corporation
Birmingham, Alabama

The cleaning of corrosion-resistant steel tubing for LOX and pneumatic service is specified by the same technique in MSFC-SPEC-10M01671 and KSC-C-123(D). A cleaning study program[1] was initiated to evaluate the cleaning process and media of these specifications and to revise the procedure as necessary to advance the present state-of-the-art of field cleaning. Level III cleanliness was proposed as a realistic goal for field systems.

Two test facilities were constructed for this program. Preliminary testing of cleaning media was accomplished with a laboratory pumping station. Clean-media that were successful in the laboratory were then evaluated with a closed-loop field pumping station. The field pumping station was designed for heavy duty service. It contained a removable 100-foot test specimen with two 30-inch sections for hydrocarbon and particle analyses in the laboratory.

Solutions were circulated through inlet 2-micron filters and returned to the storage tanks through outlet 10-micron filters. A bypass system was available to adjust fluid flow rates without involving the test specimen. Winter conditions were simulated by passing $GN_2$ through cooling coils immersed in the solutions. The test specimens and return line were cooled with an ice water bath or dry ice as required. There was a sampling port with an attached Millipore filter bomb at the downstream end of the 100-foot test specimen. The filter bomb was used to obtain particle analyses of the circulating fluids. The 30-inch sections were returned to the laboratory for an ultimate cleanliness check on the specimen itself.

Both laboratory and field tubing specimens were artifically contaminated with a mixture of 90% by weight heavy mineral oil and 10% triolein. This mixture was representative of typical lubrication compositions[2] used for working corrosion-resistant steels. The contamination mixture included 300 mg of the hydrocarbon mixture per square

---

[1]Research was supported in part by NASA, EDV-151 Division, under contract NAS8-2483. NASA Rpt. No. 10911, Vol. I Dec. 1965: Vol II Aug. 1966.

[2]Forbes, W. G., "Lubricants and Cutting Oils for Machine Tools", John Wiley & Sons, Inc., New York, New York 1943.

foot of critical surface area plus the addition of 4% of the total hydrocarbon weight as A.C. spark plug dust and salt (1:1), and 3 milligrams of red cotton linters (20-1000$\mu$ in length).

After the pumping stations were fabricated, the sequential operation of cleaning, rinsing, and purging was performed according to the cleaning procedure under study. The various media were monitored for their efficiency in removing hydrocarbons and particles during the cleaning and rinsing cycles in addition to the test sections being checked for particles and hydrocarbons remaining on the tubing after the cleaning procedure was completed.

## Analytical Procedures

Monitoring the cleaning efficiency of various procedures necessitated the validation of Test Methods A and B of MSFC-SPEC-10M01671 and KSC-C-123(D), as well as the development of an analytical procedure for the aqueous cleaning media. The nonvolatile residue (NVR) of Test Method A was limited to a maximum of 1 mg/sq. ft. of critical surface area for Level III cleanliness. Prior clean room experience with the NVR procedure had often led to negative or erratic results. Erratic results could be anticipated since the NVR residue was often less than 1 mg, and it was subjected to 220°F for approximately 4 hours.

In 1957, Istomina[3] showed that an evaluation of this gravimetric NVR method was characterized by low sensitivity and accuracy. For residues containing 3 mg or more, the error was 42%. For NVR samples of less than 3 mg, the loss of residue during treatment of the sample on a steam bath was 52%, as shown in Table 1. The observations were supported by the work of Gebhart and Reynolds[4] on the fundamentals of the evaporation of hydrocarbons. They demonstrated the Langmuir-Knudsen correlation of the proportionality of the rate of evaporation to the number of molecules on the surface, i.e., total surface area covered with molecules at any instant. The evaporation coefficient for hydrocarbons was a function of experimental conditions and not a characteristic property. In the determination of hydrocarbons in oil field waste waters, Kirschman and Pomery[5] measured the rate of hydrocarbon loss per hour during the evaporation of solvent extract on a steam bath. The average rate of hydrocarbon loss was 8 percent per hour for diethyl ether, methylene chloride, chloroform, and benzene solvents. These published findings correlated with the results obtained in this study that a minimum of a 35 percent evaporation loss could be expected and that the losses were dependent upon actual experimental parameters. The evaporation of the trichloroethylene solvent on a steam bath with a resultant nonreproducible residue was discarded as an unsatisfactory procedure.

---

[3]Istomina, K. E., Trudy Gosurdarst. Nauch. Issledovatel. i Proekt. Inst. Azot. Prom. 1957, No. 8, 248.

[4]Gebhart, H. J., Jr., and Reynolds, W. W., Am. Chem. Soc. Div. Petrol. Chem., Preprints 5, No. 3, 83 (1960).

[5]Kirschman, H. D., and Pomeroy, R., Anal. Chem. 21, 793 (1949).

TABLE 1

HYDROCARBON EVAPORATION LOSSES ON A STEAM BATH

| Investigator | Sample Size (milligram) | Average % Loss |
|---|---|---|
| Istomina | >3 | 42 |
| | <3 | 52 |
| Kirschman & Pomeroy | 1-5 | 8/hour |
| Ellenburg | 2 | 35 |
| | 1 | 54 |

Since the cleaning of the corrosion resistant steel tubing required the use of a trichloroethylene rinse, it was still necessary to find some way to determine the hydrocarbon content of the rinse. The next analytical approach was the concentration of the hydrocarbons by evaporation of the trichloroethylene at reduced pressure. This technique produced some interesting anomalies. Ten milligrams of U.S.P. mineral oil was added to 500 ml aliquots of commercially stabilized trichloroethylene. The contaminated solvent was then evaporated at 83°C and a reduced pressure of 500 mm of mercury produced by a water aspirator. The experimental apparatus was similar to Buchler system. The evaporation time was 30 minutes in contrast to the usual 4 hours that were necessary for evaporation on a steam bath. The evaporation was interrupted at about 25 ml total volume so that the samples could be analyzed by infrared differential absorption spectrophotometry. During the 30 minutes necessary to effect a 1:20 concentration of the solvent, approximately 40% of the mineral oil was lost, as noted in Table 2. The reason for the loss was the volatility of the mineral oil. All organic compounds have significant partial vapor pressures. Dalton's law that the total vapor pressure of a system is the sum of the effective partial pressures must be satisfied. During the evaporation of the samples, the ratio of the stabilizer to trichloroethylene was found to increase. The exact nature of stabilizers used in commercial trichloroethylene are proprietary; however, they are organic compounds with identifiable infrared absorption characteristics. Infrared absorption at the 2924 $cm^{-1}$ band equivalent to 19 mg of paraffinic hydrocarbons was gained from the increase in the ratio of stabilizer to trichloroethylene in these experiments. This represented an average gain of 190% over the originally added mineral oil; therefore, the two sets of errors tended to compensate each other. When the experiments were repeated, the contaminated solvent was evaporated on a steam bath for about 4 hours to a constant volume of 25 ml. There was a range of 32 to 54% loss of hydrocarbons and a gain of 16 mg from the stabilizer. These experiments again support the literature that the evaporation of solvent-hydrocarbon systems are erratic and a function of specific analytical conditions.

It was impossible to analyze quantitatively the trichloroethylene for contamination by evaporating the solvent as required in Test Method A. The next step was to attempt to find an analytical method that

TABLE 2

Variation in Composition During Evaporation of 0.05% Stabilized Trichloroethylene

| Method | Oil Added (mg) | Oil Remaining at 25 ml Total Volume (mg) | % Loss of Oil | Apparent Gain of oil From Stabilizer (mg) | % Gain |
|---|---|---|---|---|---|
| Reduced | 10 | 5.75 | 42.5 | 20 | 200 |
| Pressure | 10 | 6.00 | 40.0 | 18 | 180 |
| Steam | 10 | 4.62 | 53.8 | 15 | 150 |
| Bath | 10 | 6.17 | 38.3 | 17 | 170 |

TABLE 3

Hydrocarbon Recovery From 13X Molecular Sieve at 26°C

| Hydrocarbons Added (γ/ml trichloroethylene) | Percent Recovery |
|---|---|
| 10 | 87 |
| 10 | 91 |
| 50 | 92 |
| 50 | 86 |
| 100 | 90 |
| 100 | 92 |
| | Average 89.7 |

would use the solvent in its original state. The hydrocarbon contamination was separated from the trichloroethylene by adsorption with Type 13X molecular sieves. The sieves were eluted with carbon tetrachloride at 26°C, and the eluate was analyzed for hydrocarbon content by infrared differential analysis. The method showed 90% recovery of as little as 10 γ/ml of hydrocarbons in trichloroethylene (Table 3). The time required for the sieves to come to adsorption and desorption equilibrium was 24 hours; therefore, the method was not suitable for routine analysis.

Infrared spectrophotometry would be an ideal solution to the problem of analyzing the contaminated trichloroethylene. Only about 45 minutes of analytical time would be required per sample, and no laboratory manipulations would result in the loss of hydrocarbons. Missile degreasing grade trichloroethylene, by differential analysis, was analyzed in the "as received" condition to a lower limit of sensitivity of 150 micrograms of hydrocarbons per milliliter of solvent with a

Perkin-Elmer Model 237 infrared spectrophotometer. This arbitrary lower limit of sensitivity was imposed by the loss of differentiation of the signal-to-noise ratio of the hydrocarbon infrared absorption peak at 2924 $cm^{-1}$ imposed over a major absorption peak of the stabilizer present in the trichloroethylene. The signal-to-noise ratio was improved by the use of a scale expansion accessory to expand a selected portion of the total radiant energy output of the instrument. In this case, it was possible to expand the instrument output between 72 and 82% transmission at 3000 to 2900 $cm^{-1}$ to lower the limit of sensitivity of the infrared differential analysis to 10 micrograms of hydrocarbons per milliliter of stabilized trichloroethylene, as shown in Table 4. The absorbances followed Beer's Law and had satisfactory precision.

TABLE 4

Infrared Analysis of Trichloroethylene
(Differential Absorption and Scale Expansion)

| Hydrocarbons ($\gamma$/ml of trichloroethylene) | Log $I_o/I$ at 2924 $cm^{-1}$ |
|---|---|
| 150 | 0.274 ; 0.275 |
| 100 | 0.176 ; 0.177 |
| 50 | 0.095 ; 0.097 |
| 10 | 0.024 ; 0.021 |

Scale expansion provided a suitable method for the infrared analysis of the bulk quantities of trichloroethylene that were to be used in the field pumping station. However, the test sections of cleaned tubing were checked in the laboratory after the cleaning procedure was completed. If they were rinsed with a solvent having solvency capabilities equivalent to trichloroethylene, but not having carbon-hydrogen bonding to interfere with the 2924 $cm^{-1}$ infrared band, the limit of sensitivity for detecting hydrocarbons could be significantly lowered. The lack of carbon-hydrogen bond in the molecule limited the solvents to carbon tetrachloride and trichlorotrifluoroethane.

A comparison of solvent properties as listed in Table 5. Carbon tetrachloride whould be selected on the basis of the comparison of the Kauri-butanol values, which are a measure of the solvent efficiency of a material evaluated on a volume basis. The fluorocarbon solvent would be selected on the basis of personnel safety. Since these checks were to be performed by qualified laboratory personnel in a well exhausted hood, the concern for safety was no greater than that for normal laboratory operations. In addition, the carbon tetrachloride had a higher boiling point which approximated that of trichloroethylene.

Type 304 stainless steel panels, 3-1/4 by 1-1/4 inches, were coated on one side with 10 mg of the contamination oil mixture. A panel was then rinsed with 1 ml increments of one solvent for four successive times. Each rinsing was collected separately and analyzed for total hydrocarbon content by infrared differential analysis. The average removal rate of 7 tests for each solvent is shown in Table 6. The distribution coefficient for the fluoronated hydrocarbon was 0.85 while that

of carbon tetrachloride was 0.98. The actual minimum volumes of solvent necessary to effect these distribution coefficients were in the ratio of 1 volume carbon tetrachloride to 4 volumes fluorocarbon. These results were supported by the same approximate ratio of Kauri-butanol values. Material balance studies of rinsing contaminated tubing with carbon tetrachloride showed that 99.5 per cent of the contamination was recovered from the tubing.

TABLE 5

COMPARISON OF SOLVENT PROPERTIES

| Compound | Maximum Boiling Pt. $^{o}F$ | Kauri-Butanol Value | Toxicity (ppm by volume) |
|---|---|---|---|
| $C_2H_2Cl_3$ | 190 | 130 | 200 |
| $CCl_4$ | 172 | 114 | 25 |
| $C_2Cl_3F_3$ | 118 | 31 | 1000 |

TABLE 6

RESULTS OF PANEL DEGREASING TESTS[a]

| Rinse No. | Mg Oil Removed Per Rinse (avg. 7 tests) | |
|---|---|---|
| | $C_2Cl_3F_3$ | $CCl_4$ |
| 1 | 8.50 | 9.81 |
| 2 | 1.10 | 0.18 |
| 3 | 0.24 | N.D. < 0.01 |
| 4 | 0.13 | N.D. < 0.01 |
| Total | 9.97 | 9.99 |

(a) 10 mg oil on 3-1/4 by 1-1/4 inches Type 304 steel panels

The investigations of Rather[6], Lecomte[7], and Simard[8] had all shown that the determination of total hydrocarbon matter in refinery effluent water was best accomplished by extracting the waste water with carbon tetrachloride and quantitatively determining the hydrocarbons by infrared absorption at 3.5 to 3.38 microns. Therefore, the final analytical technique for this program consisted of using carbon tetrachloride as the extraction medium for monitoring the aqueous solutions and as the flushing solvent for the test sections used to determine hydrocarbon residues. Since the carbon tetrachloride was required to be of spectrophotometric grade, cost considerations dictated the use of the fluorocarbon solvent or trichloroethylene for the larger flushing volumes necessary for particle analyses. Hydrocarbons were determined by infrared differential analysis at 2924 $cm^{-1}$ with a Perkin-Elmer Model 237 double-beam automatic spectrophotometer. The absorbances followed Beer's Law and the overall analytical method had 99.5 percent recovery of hydrocarbons in the 1 to 10 milligram range. Typical data for ten complete analyses showed excellent precision with a coefficient of variation of 0.5.

Particle populations were determined as directed by Test Method A and Level III cleanliness of MSFC-SPEC-10M01671. While the manual counting of particles leaves much to be desired, no commercially available automatic particle counters appear to be able to perform the task with precision and accuracy and at reasonable cost. Laboratory technicians trained until they were able reproducibly to count standard filters seemed to be the best solution of the problem for this program. There were over 15,000 particle counts consisting of samples of circulating fluids taken in the field with Millipore filter bombs and those of the cleaned tubing taken in the laboratory by solvent flush. The filter bombs were limited to manual counting. There is some interest in a commercially developed television-microscope combination for counting filters. The projected television image of the filter can then be electronically scanned. However, this instrumentation has not yet progressed to being a useful tool.

The volume of solvent to flush cleaned tubing should be maintained at 500 ml/sq. ft. The reduction of volume to 100 ml/sq. ft. that is allowable in MSFC-SPEC-10M01671 for larger parts was unsatisfactory, as shown in Table 7. Several 2-inch diameter tubes were contaminated, cleaned, and then checked with successive 300 ml portions of solvent. Each particle count was an average of 7 tests. While this was not extensive testing, it was indicative of a situation that could not be ignored. The larger volume of solvent was particularly necessary for the fluorocarbon solvents with their lower solvency power since it was first necessary to solvate any hydrocarbon "glue" that held particles to the wall of the tubing before they could be rinsed out.

---

[6]Rather, J. B., Jr., et. al., Anal. Chem. 30, 36 (1958).

[7]Lecomte, J., Bull. soc. chim. France 1949, 923.

[8]Simard, R. G., Hasegawa, T., Bandaruk, W., and Headington, C. E., Anal. Chem. 23, 1384 (1951).

TABLE 7

Particle Removal From Three Square Feet Surface Area

| Rinse No. | Volume Used (ml) | Average Particle Count[(a)] > 35 Microns | |
|---|---|---|---|
| | | $C_2H_2Cl_3$ | $C_2F_3Cl_3$ |
| 1 | 300 | 82 | 55 |
| 2 | 300 | 31 | 26 |
| 3 | 300 | 10 | 32 |
| 4 | 300 | 5 | 27 |
| 5 | 300 | 2 | 18 |

(a) Series of 7 tests

The following conclusions can be drawn from this study:

1. The NVR procedure is an unreliable method of analysis. This unreliability is the result of a thermodynamically inherent error since the effective partial vapor pressures of the organic constituents cannot be eliminated.

2. Infrared differential spectrophotometric analysis of the solvent test solution is a reproducible and precise substitute method of analysis.

3. The infrared technique is simple enough to be adapted to on-stream automated control with existing present state-of-the-art equipment.

4. Fluorocarbon solvents cannot be indiscriminately substituted for trichloroethylene in Test Method A since they have less capability for dissolving hydrocarbons. They require a comprehensive study and revision of the test procedure before use.

5. The minimum value of 500 ml of solvent per square foot of critical surface area is essential for effective particle removal even for large parts.

## Cleaning Studies

Once the analytical procedures were determined, Procedure 3 of MSFC-SPEC-10M01671 for cleaning corrosion-resistant steel tubing was evaluated. It was soon evident that the excessively high solution temperature and the highly alkaline trisodium phosphate specified in Procedure 3 created numerous technical and economic problems. Various new cleaning media and procedures were then investigated in order to develop a new procedure for cleaning corrosion-resistant steel tubing in closed-loop systems.

During the course of this investigation, 9,300 linear feet of tubing was cleaned by using various formulations in the field pumping station. The tubing could be field cleaned to Level III cleanliness under diverse environmental conditions. The following generalizations were developed from the reduction of this data:

1. Particles and fibers were removed more efficiently by the cleaning cycles than by the rinsing cycles. Cleaning removes approximately 2 to 5 times as many particles and fibers as rinsing.

2. The rinsing cycles show a maximum in particle and fiber removal 5 to 10 minutes after the start of the filtered tap water rinse.

3. The first 10 minutes of the filtered tap water rinse removed at least 90% of the hydrocarbons.

4. The rinseability of surfactants and surfactant-soil micelles are temperature sensitive in closed-loop circulating system. Increased temperature results in increased rinseability. At any given temperature, low foaming surfactants rinse more easily than moderate or high foaming surfactants.

5. The particle content of circulating media is not necessarily a measure of the final level of cleanliness of the system. Each system must be evaluated under anticipated operating conditions.

6. The final hydrocarbon level of cleanliness of a system can be related to the angle formed by the cleaning solution in contact with the contaminated metal surface. This equation is

$$C = k \tan \theta$$

where c = hydrocarbon residue remaining after cleaning in mg/sq. ft.
k = experimental constant for the system
$\theta$ = contact angle

## Velocity Studies

The evaluation of the $GN_2$ purge of Test Method B of MSFC-SPEC-10M01671 was requested in the original work statement of this study. The 7 SCFM rate of Test Method B was to be compared to a maximum feasible $GN_2$ velocity purge for the same time after the tubing specimens were cleaned and dried. Test Method B required a $GN_2$ purge of 7 SCFM for 3 minutes and a 0.45$\mu$ Millipore filter downstream of the test specimen to retain the dislodged particles. This low flow rate of $GN_2$ probably did not dislodge all of the particles remaining in the tubing after cleaning. To test the validity of this assumption, a maximum feasible velocity $GN_2$ purge for 3 minutes was used immediately after Test Method B. The maximum flow rate attainable without extensive

alterations in the present cleaning system was 35 SCFM at 350 psig. In addition to counting the particles retained by the Millipore filters from Test Method B and the maximum feasible velocity test, the 30-inch test section was returned to the laboratory for an additional particle check. The test section was rinsed with solvent and the particles checked as usual by Test Method A. Thus, the test section provided an additional check on the effectiveness of the 35 SCFM purge for dislodging particles. Although the testing by Test Method B was satisfactory to Level III particle population, the additional 3 minute maximum velocity purge always dislodged almost three times as many more particles and fibers as shown in Table 8. After extensive testing of cleaned tubing, it was evident that the nitrogen velocity of Test Method B was not sufficient to provide an adequate particle population check. At this point in the study, an additional 0.45$\mu$ Millipore filter was added immediately upstream of the specimen. This eliminated the necessity of a blank count on the purging nitrogen. It also provided assurance that all particles retained by the downstream filter were generated by the specimen.

TABLE 8

Typical Evaluation of the Effectiveness of Test Method B

| Tubing Size (Inches) | Contamination Range (microns) | Test Method B | 3 min Max Velocity Purge | Test Method A After Purges |
|---|---|---|---|---|
| | Particles | | | |
| 3/8 | 0-35 | 263 | 567 | 15 |
| | 36-60 | 6 | 13 | 12 |
| | 61-95 | 0 | 4 | 2 |
| | 96-135 | 1 | 0 | 1 |
| | 136-170 | 1 | 2 | 0 |
| | 171-350 | 1 | 3 | 0 |
| | 350+ | 0 | 5 | 1 |
| | Fibers | | | |
| | 0-35 | 0 | 10 | 2 |
| | 36-350 | 2 | 10 | 5 |
| | 351-700 | 1 | 5 | 2 |
| | 701+ | 0 | 3 | 2 |
| | Particles | | | |
| 1-1/2 | 0-35 | 100 | 275 | 20 |
| | 36-60 | 2 | 12 | 14 |
| | 61-95 | 0 | 6 | 5 |
| | 96-135 | 0 | 0 | 5 |
| | 136-170 | 0 | 1 | 0 |
| | 171-350 | 0 | 0 | 1 |
| | 350+ | 0 | 2 | 0 |
| | Fibers | | | |
| | 0-35 | 4 | 2 | 5 |
| | 36-350 | 8 | 13 | 11 |
| | 351-700 | 0 | 0 | 0 |
| | 701+ | 0 | 2 | 1 |

The variation of $GN_2$ velocity with tubing size could be calculated to a first approximation. By combining the equation of Continuity and the Perfect Gas Law, a useful equation for the velocity of the nitrogen was derived in terms of quantities that could be determined during actual testing:

$$V = \frac{0.0849\ QT}{Pd^2}$$

where

V = velocity in ft/sec
Q = flow in SCFM at exit of 100 foot test specimens
T = temperature in or at point of measurement
P = pressure in psia at point of measurement
d = inside diameter of tube in inches

A reasonable approximation of the variation in actual tubing velocities could then be obtained by measuring the temperature and pressure of the gas at points located 10 and 90 feet downstream of the beginning of the test specimen. Calculations were made for exit velocities of 5, 10, 15, 20, 30, and 40 SCFM. These experiments showed some interesting anomalies, as presented in Table 9. By the time the tubing diameter had increased to 1 inch, the exit gas velocity had had decreased by a factor of 20. The velocity had decreased by a factor of 80 with 2-inch tubing. This variable is not recognized in Test Method B, and it is one reason why the test is not reliable.

TABLE 9

Observed Tubing Velocities

| Tubing Size (inch) | Flow Rate (SCFM) | Tubing Inlet Velocity (ft/sec) | Tubing Outlet Velocity (ft/sec) |
|---|---|---|---|
| 1/4 | 5 | 44.0 | 80.5 |
| | 30 | 81.8 | 166.0 |
| 3/8 | 5 | 30.5 | 35.6 |
| | 30 | 70.5 | 87.0 |
| 1 | 5 | 3.87 | 3.92 |
| | 30 | 9.5 | 9.6 |
| 2 | 5 | 1.035 | 1.035 |
| | 30 | 2.60 | 2.66 |

Preliminary experiments with purging the cleaned tubing had indicated that a longer purge time might eliminate most of the particles. A new procedure was initiated for the remaining 80 experiments. After the tubing was cleaned and dried, it was

1. Checked by Test Method B
2. Checked by 3-minute maximum feasible velocity purge
3. Purged for 30 minutes at maximum feasible velocity
4. Rechecked with 3-minute maximum feasible velocity purge
5. Checked in laboratory by Test Method A (30-inch test section).

Typical results of the total counts of these experiments are shown in Table 10. The following conclusions may be drawn from these experiments:

1. Test Method B was not an effective check for particle population.
2. Gaseous purging of a system after cleaning was advantageous in removing particulate contamination. The minimum effective parameters for dislodging particles were a 30-minute purge of $GN_2$ at a minimum flow of 35 SCFM in 2-inch or smaller diameter tubing. A higher flow rate of $GN_2$ with the resulting greater turbulence within the tubing should be used whenever possible.
3. A more meaningful gaseous test method would consist of a 10-minute purge with a specified minimum velocity, expressed in ft/sec, determined at the point where the filter is located for collecting the particles. A recommended minimum velocity would be 10 ft/sec.
4. The use of Test Method A (liquid flush) to check the particle population was preferred rather than any type of $GN_2$ purge if it is feasible to adapt Method A to the system being checked.
5. A satisfactory Level III particle check by Test Method A on the 30-inch test section used in this Cleaning Study Program was representative of the level of cleanliness of the entire 100-foot test specimen.

There was no direct correlation between the velocity of the $GN_2$ and the total number of particles removed by purging the tubing cleaned in this study. However, there was an uncontrolled variable in the experiments since the tubing was already cleaned before purging. The total number and size distribution of the particles remaining after cleaning were purely chance. Additional experiments would be necessary to ascertain the maximum particle size of a specific density that could be moved by specific velocities, as well as to determine what correlation existed between total particles dislodged and the velocity of the purging gas. The removal of hydrocarbons from tubing by purging is another problem that must be discussed another time. However, it is sufficient to note that anomalies also exist in this field and that this technique merits extensive investigation.

TABLE 10

Test Method B and Maximum Feasible Velocity Particle Checks

| Exp | Blend | Tubing Size (inches) | Test Method B Total Count | | 1st 3-Minute Max Velocity Purge Total Count | | 30-Minute Max Velocity Purge Total Count | | 2nd 3-Minute Max Velocity Purge Total Count | | Test Method A 30" Test Section | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Particles | Fibers | Particles | Fibers | Particles | Fibers | Particles | Fibers | Particles | Fibers |
| 175 | VII | 1 | 1264 | 34 | 1820 | 21 | 3838 | 31 | 492 | 2 | 61 | 17 |
| 182 | VII | 2 | 157 | 10 | 1480 | 25 | 3100 | 25 | 505 | 9 | 539 | 23 |
| 184 | VII | 3/8 | 81 | 1 | 535 | 7 | 776 | 25 | 233 | 8 | 45 | 12 |
| 189 | V A | 1/4 | 2494 | 22 | 279 | 15 | 6181 | 16 | 815 | 1 | 220 | 10 |
| 193 | V A | 1-1/2 | 1590 | 21 | 3908 | 21 | 7512 | 23 | 1203 | 10 | 721 | 3 |
| 197 | V A | 1 | 2139 | 6 | 3202 | 21 | 6561 | 32 | 1818 | 22 | 630 | 7 |
| 201 | X | 1 | 8231 | 19 | 9731 | 18 | 8933 | 12 | 3023 | 4 | 214 | 4 |
| 204 | X | 2 | 836 | 29 | 1111 | 7 | 2939 | 50 | 754 | 8 | 90 | 10 |
| 206 | X | 3/8 | 970 | 20 | 916 | 13 | 869 | 9 | 363 | 7 | 91 | 14 |
| 213 | X A | 2 | 867 | 25 | 1139 | 25 | 5447 | 52 | 1365 | 13 | 818 | 13 |
| 214 | X A | 3/8 | 859 | 22 | 3229 | 18 | 5461 | 77 | 1527 | 25 | 923 | 13 |
| 219 | X A | 1-1/2 | 713 | 9 | 1434 | 19 | - | - | - | - | 516 | 9 |

Summary

Monitoring the cleaning efficiency of various procedures necessitated the validation of Test Methods A and B. Both methods, per se, proved to be inaccurate. Evaporation of the test solvent of Method A for nonvolatile residue content was erratic. This procedure incorporated a thermodynamically inherent error since effective partial vapor pressures of the organic constituents could not be ignored. Infrared differential absorption spectrophotometric analysis of the solvent test solution provided a reproducible and accurate substitute method of analysis. Other sophisticated analytical methods of instrumental analysis, such as gas chromatography, could be incorporated into the monitoring system. For Test Method B, extensive development is necessary to correlate purging parameters with the intrinsic volatility characteristics of contaminating hydrocarbons, and with the size and specific density of particles. In conclusion, it must be emphasized that there is a great gap between recognizing that a test method is inadequate and defining the basic theoretical parameters necessary to upgrade that test to a meaningful and accurate method.

# 4. AUTOMATED FINAL CLEANING, RINSING, AND DRYING STATION

by H. V. Anthony, J. P. McDonald, M. Piccone

Presented by

J. P. McDONALD

Martin Marietta Corporation
Denver, Colorado

## Introduction

All existing methods for final cleaning, rinsing, and drying of aerospace vehicle fluid system components require a rinse and a transfer of the sampling fluid to another position or location for analysis. Aerospace industries have relied on criteria that have not been dimensionally defined, at least in the areas of force of impingement, velocity, orientation of the surface being sampled, accessory sampling energy, and other characteristics that vitally affect the validity of results. Due to this lack of definition, the results have been almost totally dependent on operator interpretation. As a result, reproducibility of final rinse results and hence specification conformance have been inconsistent from program to program and, in some cases, from component to component.

This paper discusses a means of completely quantifying and automating the evaluation rinsing, sample collection, and sample preparation (filtration, evaporation, drying, etc.) operations necessary for the qualification of hardware to aerospace vehicle fluid system cleanliness criteria for particle and nonvolatile residue content.

In addition to providing this unique capability, the equipment discussed would permit all of the operations from final cleaning through sealing to be performed in a normal factory environment. This last ability is not essentially novel, but the elimination of the requirement to perform separate manual evaluation rinses and to remove the effluents to another location for analysis would provide a new dimension of practicality.

Thus, commercially clean hardware could be placed in a chamber and, with no further handling, be removed as qualified hardware into a clean environment for assembly or sealing with no delays due to laboratory support activities, or the extra rinsing and processing preparatory to these support activities.

Adoption of the method would offer significant savings in personnel time and handling costs. However, the convenience and cost-savings factors are clearly secondary to increased reliability of sampling and analysis, reduced number of rejects, the use of much more stringent particle level criteria, and the capability of defining and programming the exact evaluation rinsing conditions.

## Soluble Versus Particulate Contamination

One can develop many classification systems of the sampling methods, analytical methods, and tests (including visual inspections) that are applicable to the contamination control of fluid-conducting surfaces and operational or service fluids that constitute Aerospace Vehicle and Spacecraft Operational Fluid Systems. Probably in initial, fundamental division is the separation of the contaminant being evaluated into its nature of occurrence; that is, whether it is particulate contamination or is essentially fluid contamination. The latter category includes contaminants dissolved in the flush or operational fluid, as well as colloids, sols, gels and particulate suspensions which are not filterable by the membranes specified for particulate analysis and which are invisible as a separate phase in the specified methods of examination of the fluid.

Sampling of fluid or condensable gaseous contaminants from hardware is usually performed by a solvent flush which has a known solvency for the contaminants of interest or by absorption or absorption on a media through which a gaseous test fluid effluent from the surfaces being evaluated is passed.

The flow rates and input energy conditions, other than temperature, usually are not critical to the sampling operation for the reason that the solvent and the time of contact will usually assure that a homogenous distribution of the soluble contaminant will be achieved in the effluent, or that a sufficient indication will be achieved to flunk the criteria. The condensable gaseous contaminants are commonly assumed to be homogenously distributed in the gaseous test media by molecular diffusion and by the nominal turbulence of minimal sampling flow rates so that any portion of the effluent may be regarded as an aliquot. Indeed, it is common practice to limit severely the flow rates and pressure in the latter determination and to require a quiescent soak time to ensure a representative or significant indication of the gaseous condensables. Thus severe energy inputs in the sampling process (other than temperature) are usually not necessary, and are sometimes regarded as detrimental to the process.

These considerations for hardware are based also upon the premise that fluid or dissolved contaminants in the test medium are normally residual -- that is, they were built into the hardware in the act of manufacture and will not accrue by transport into the system in the operational fluids. Also, they will not ordinarily be generated by the system itself once it is closed up and experience only operational or blanketing fluid for the remainder of the operational period (before system entry for modification, servicing, or component replacement).

The considerations of optimum sampling parameters for particulate contaminants, however, dictate the use of a diametrically opposed approach for this operation. For particulate sampling, temperature is not important since molecular diffusion is not involved; also, the maximum mechanical energy input conditions attendant to the process that can be practicably achieved are desired. Efforts are made to tap the system with hammers during sampling, vibrate it with air hammers or electric vibrators, produce cryogenic shock by filling it with cryogenic fluids, produce maximum turbulence by high-velocity test fluid flow (through as large a sampling orifice as possible), and deliberately slam fast-acting valves open and closed to produce hydrostatic or inertial shock.

Why? Because it is known that mechanical energy is the only efficient way of dislodging particles and fibers to permit their removal. Whereas the migration of the fluid contaminants is caused by their own molecular and atomic energies, all of the motivating energy for the diffusion and transport of particulates must come from sources exterior to the contaminants.

In the size-ranges and types of particulates normally encountered, the chief concern is with frictional or mechanical entrapment of particle matter existent prior to sampling and with materials of construction which become particulates during normal operational life of the system. The former category includes: casting sand, abrasive blasting media and machining tool fragments embedded in metallics, inclusions in elastomers, and foreign particles cemented by corrosion products or processing fluid residues. The latter category includes in-place corrosion products, heat treat scale, weld scale, deteriorating conversion coatings, plating or nonmetallic coatings, and fragments of the construction materials. These fragments are generated by shearing, spalling, friction, and fracture due to impact and abrasion, structural stress, chattering, vibration, thermal and hydraulic shock, hydrostatic pressure, or by any conceivable change of potential-to-kinetic energy in the system during operation.

The facts are that systems designed with even the most compatible materials and sound engineering principles wear out. They wear out by distortion of or loss of material, some of which moves downstream as particulates in the operational fluid.

Although this function is cumulative and partially time-dependent with respect to the aging characteristics of the materials subjected to daily and seasonal variation of temperature, operational cycles, seismic shock, etc. a representative indication of the particle generating potential of systems can best be obtained by an accelerated, vigorous energy-input schedule during the evaluation purge or flush for particulates.

This operation is intended to knock the obstinate particles loose from significant surfaces and produce that efflux of particulates, qualitatively and quantitatively representative of those which will be spawned by the system in service.

As already stated the sampling needs of "soluble" contaminants are well provided for by the selection of suitable solvents or miscible gases and by temperature regulation. This is true for the significant surfaces of details and assemblies and for operational fluids.

For particulates however, although some attention is given to suitable kinetic energy inputs for operational fluid and for system hardware sampling, the parameters of sampling of particles and fibers at the detail part level are totally neglected unless the part is a conducting component, such as a hose subject to minimum sampling flow rates, or a tank subject to defined impingement and flush procedures.

It is also important to consider that the components for which no energy input requirements are defined (that is essentially monolithic details with exterior or partially exposed and unshielded significant surfaces) comprise probably in excess of 98% of the flyable hardware and substantially all of the dynamic hardware -- that is

surfaces which are required to move differentially to perform their required function in flight. Outside of orifice plugging considerations then, these latter components are the very ones that we can least afford particle contamination on. They include critical sliding and rolling surfaces of valves, pumps, regulators, and actuators which take the most mechanical punishment in flight and during transfer operations in the ground support equipment.

The failure to supply energy-input requirements for sampling parameters for detail parts and components is a very serious error. For assembled components and subsystems, it is recommended that these be sampled for particulates only when they are experiencing the total spectrum and amplitude of launch and flight vibration (or worst-service impact for ground equipment) and while they are simultaneously being flowed with test fluids that adequately simulate the physical and chemical forces of operational flow. In most instances, this is the operational fluid itself. In addition effluents should be examined in total for particulate content, or representatively sampled by means of turbulence-producing or isokinetic sampling devices, and processed for analysis by microscopic examination of membrane-filtered deposits.

For detail parts, the solution to the problem is for the industry to adopt automated final rinse methods in a cabinet-type operation, with programmed, universally established energy inputs, automatic processing of effluent for determination of particulates, and possibly, automatic processing of effluent for fluid contaminant determination.

Some of the Automatic Final Cleaning Cabinet manufacturers have taken steps to integrate automatic particle monitoring devices in the fluid streams of their equipment. As yet, however, there is no indication that any of them have placed this capability where it will be most significant, in the effluent stream from the chamber.

Also, none has provided the capability for microscopic examination of membrane-filtered effluent particulates as a portion of the device. This capability is vital because the diagnostic and qualitative information afforded by microscopic observation of the filtered particulates cannot be obtained from any automated counting machines existent or conceptual. The zone-sensing devices for counting particles in fluids provide information for flowing operational fluid streams that microscopic methods cannot, since the microscopic method is necessarily a batch type sampling device. In this application however, this limitation does not apply since the automated final rinse cabinet operation is also a batch method and the entire effluent from it may conveniently and practicably be routed through the membrane. The In Line Filter Holder and Counter (ILFHC) is regarded as an important portion of this concept.

## In Line Filter Holder And Counter

Figure 1 shows an earlier configuration of the ILFHC proposed specifically for automated Final Rinse application. It is designed for extremely rapid replacement of the filter medium, low hydrostatic pressure application, and simplicity of operation. These are oriented toward its intended operation in a semi-fixed, sheltered location on a fast, repetitive cycle.

The sampling fluid stream enters the apparatus through the upper flexible tubing (A), which may be any inert, nonfriable, abrasion-resistant plastic such as Teflon, Tygon, or Kel-F. This tubing must be sufficiently long to permit the rotatable portion of the assembly (all portions except mounting bracket (0) and the microscope attached to it) to rotate on bearings (M) about the axis in an arc of 180 degrees in either direction from the neutral position without collapsing the tubing.

The sampling stream is then conducted through the inlet tube (B) into the upper filtration chamber and through the filter (D) supported on the backup screens (E, F). The fluid then passes out of the assembly through the outlet tube (H) and is returned to the source system through the flexible tube (I).

Note that fluid flow through the filter (D) is permitted only through an annular area with an inside diameter governed by an impervious concentric circular area (F) in the backup screen (E) and an outside diameter governed by either the chamber walls or by a similar impervious area in the backup screen (E) as required by the microscopic field.

The chamber is dried by valving-off the sample fluid stream upstream of inlet tube (B) and downstream of outlet tube (H) and introducing a vacuum through (H) sufficient to cause the vapor pressure of the fluid being sampled to exceed the absolute gas pressure in the filtration chamber.

An alternative method is to purge the filtration chamber by introducing a clean, dry inert gas such as nitrogen into the inlet tube (B) to carry the sample fluid vapor and liquid residue through the outlet tube (H).

The particle matter retained on the filter (D) is examined, counted, and sized by means of the long-focal-length, approximately 50-power angle microscope (T, P, S), which views the material through the optical window (C). The window forms an optical quality glass top to the filtration/counting chamber.

The microscope is provided with a traversing mechanism (Q, R, V) that provides controlled linear movement in at least one horizontal direction in a plane coincident with the axis of the total assembly so the movement of the objective tube (S) traverses the diametric lines of the filter (D). The crank arm and screw (V) turning through the fixed, matching, internally threaded opening in the bracket (Q) moves the yoke (R) to which the microscope (P) is horizontally gimbaled.

The ability of the rotatable portion of the assembly to rotate 180 degrees in either direction from the neutral position, coupled with the traversing capability of the microscope, allows all portions of the filtration surface to be microscopically examined.

Used filters are replaced by pulling vertically downward on the outlet tube (H), thus compressing the spring (J) and allowing access to the filter in the opening between the seal plate (N) and the lower chamber elements (G, E, F). The spring retainer (L) is sufficiently slotted vertically to allow manual access to the filter and backup screen when the elements of the lower chamber (G, E, F) are depressed.

During lowering and resealing, the guide rods (K), which pass through guide holes in the lower chamber wall (G), maintain the concentric and azimuth alignment of the lower chamber with the upper chamber.

Since the spring (J) must exert sufficient force to effect a fluid-tight seal between the lower chamber elements and the seal plate and attached elastomer seal (N), mechanical aids can be added to assist in compressing this spring during membrane exchange (e.g., a foot pedal mechanically linked to the outlet tube (H) or the lower filtration chamber elements).

By means of this tool a quantity of the fluid being sampled for particles may be passed through the assembly and the final rinse chamber. A preliminary particle count of the filter may then be made, by the methods previously described, to determine background contamination. The fluid being sampled is again flowed through the assembly for a controlled total volume of flow, the chamber is again vacuum-dried or dried by a clean inert gas purge, and a second particle size and count made. The size and count data obtained from the initial filtration are subtracted from those obtained from the second filtration, thus eliminating the particle contribution from all sources except that suspended in the sample stream. The preliminary equipment flush and count may be eliminated when the background contamination is not significant.

## In-Line Nonvolatile Residue Sampler Evaporator

Figure 2 shows a concept of an automated Nonvolatile Residue sample collecting and processing device. In this form, automatic readout is not illustrated although it could be provided.

Since this configuration was proposed further investigation has disclosed that the vacuum-jacketed Cahn electrobalance offers ready adaptability to completely automated NVR sampling, analysis, readout and recording on either a drop-wise or milliliter sample quantity basis in cycle times less than a minute.

Recent literature references indicate that some new types of automatic NVR analysis machines for on-stream use have been developed. However, there seems to be some ambiguity in the term NVR. In the past some commercial devices have been referred to as NVR removal or processing devices whereas a closer inspection revealed that they were capable only of mechanical filtration and hence unable to do anything about the nonvolatile soluble contaminants which often constitute the substantial total of the NVR. In any event any satisfactory NVR analysis device can be used in this application.

The cycling principle of the device shown in Figure 2 is described below.

The fluid to be sampled leaves the process or operating fluid stream (A) at supply port (B) and is conducted in a freely falling stream by an inert, nonfriable, abrasion-resistant, plastic flexible tubing (C) such as Teflon, Tygon, or Kel-F through the chamber inlet

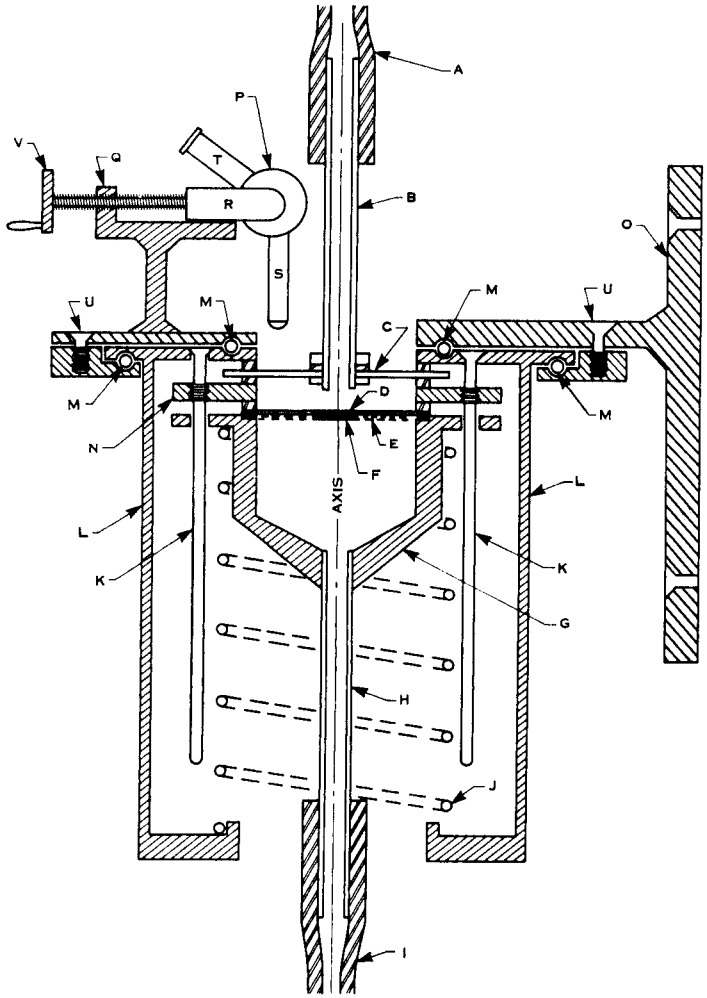



Figure 1. In-line filter holder/counter

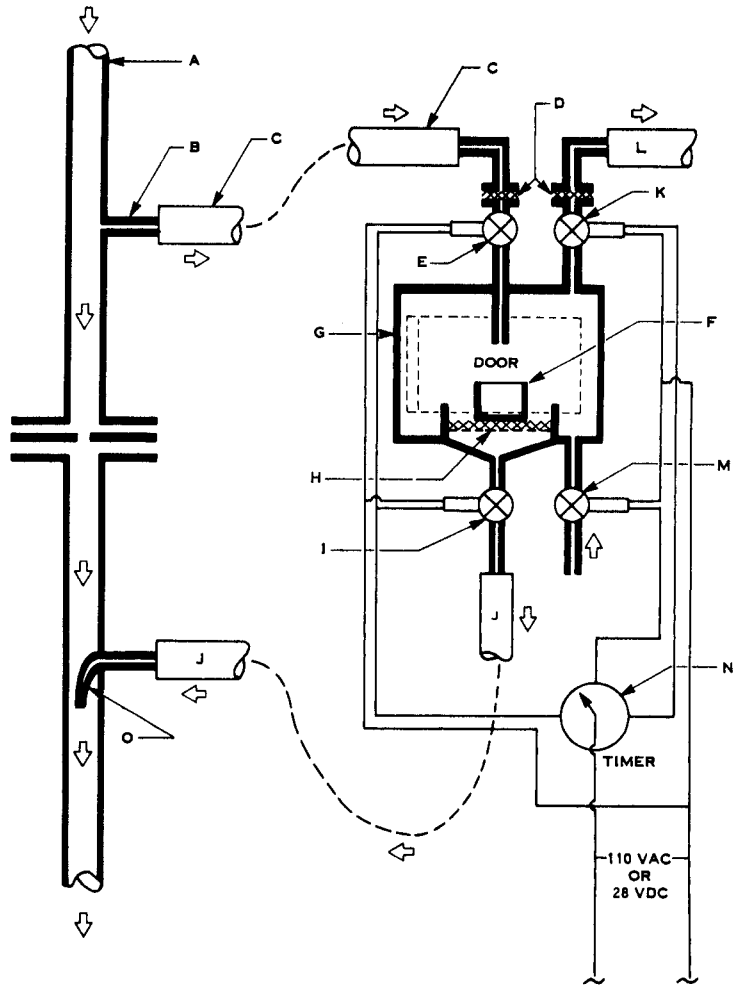



Figure 2. In-line nonvolatile residue sampler/evaporator

tube, filter orifice (D), and solenoid valve (E) into the NVR cup (F). The configuration and capacity of this cup are critical to the concept only in the capability of entrapping and retaining a known volume of fluid and reproducing the process under repeated cycling of the apparatus.

The overflow from the cup drains through the support screen (H), through the solenoid drain valve (I), through the flexible tubing (J), to the aspirator (O). The flexible tubing (J) has characteristics identical to those of the inlet flexible tubing (C).

The amount of suction produced in relation to the flow of stream (A) can be adjusted either by rotating the aspirator (O) about an axis perpendicular to the axis of the fluid stream (A), or by using an adequate metering valve.

The rate of sample fluid stream flow through the NVR chamber (G) must be great enough that the NVR cup (F) overflows during one cycle and small enough that the NVR cup does not overflow from the force of impingement (sloshing).

The height of the inlet nozzle to the chamber (from valve E) above the level of the liquid in the NVR cup is not critical and it may extend to the liquid level to prevent spattering.

Valves (E, I) are programmed shut by timer (N) and valve (K) is opened to vacuum line (L), which conducts to a vacuum source capable of evacuating the NVR chamber (G) to an absolute pressure less than the vapor pressure of the fluid being sampled at the sampling temperature.

The rate of vacuum application is controlled by orifice (D) in the vacuum line so that the chamber evacuation rate is not sufficient to cause the NVR cup to boil over. Since more rapid boiling of the sample fluid in the NVR cup is permissible as the level of liquid in the cup falls because of the increased "free-board," the evaporation is initiated at a very slow rate and proceeds to a quite vigorous termination point. Timer (N) then closes valve (K) and opens valve (M), which readmits atmosphere to the NVR chamber (G).

The door (shown in phantom view in Figure 2) is then opened and the now dry NVR cup (F) is removed by forceps to the pan of an analytical balance for weighing. The difference between this weight and the cup's precycle weight factored by the volume of the cup or the weight of liquid entrapped in the cup (determined by multiplying the volume of the cup by the density of the sampled fluid) gives the NVR of the sampled fluid. The NVR cup is then replaced in the NVR chamber and the sampling cycle is complete.

The timer (N) may be appended to the apparatus as shown, or it may be the main sequencing timer of a commercial automatic final cleaning/rinsing/drying machine for which the in-line NVR sampler/evaporator is expected to be an accessory. Under the latter condition, the in-line NVR sampler/evaporator will sample both the influent and effluent processing streams of the final cleaning/rinsing chambers of the commercial machine, or will sample the effluent processing stream alone. It also will, in conjunction with the in-line filter holder/counter, be used as an accessory to the commercial machine to permit this machine to be used as an automated cleanliness testing machine.

With present methods, it is necessary to draw a liquid sample; transport it to a heated surface, bath, vacuum chamber, or some combination of the three; cool the container in a desiccator; and reweigh the sample.

The in-line NVR sampler/evaporator eliminates the extra liquid handling and transfer operations by catching the sample and boiling it off directly. Increased reliability is inherent in the reduction of surfaces that the sample and NVR container touch during processing, and there is a corresponding time saving.

Whatever the configuration, at least a portion of the chamber (probably the door) should be transparent so filling, draining, and evaporation may be observed without unsealing the chamber.

To update this concept to current technology, chamber G would be the hangdown tube of the Cahn electrobalance and the sample inlet valve solenoid E would receive its closing signal from the electrobalance output instead of from the timer. The Cahn electrobalances are extremely sensitive and rugged instruments which can be programmed for fast sensitivity and capacity changes. Consequently the timer will place the balance in the high capacity-low sensitivity mode for the sample influx operation (which will now be weight-measured rather than volume-measured by the means already described) and will then program to high sensitivity for weight of the residue after the evaporation step. Decimal proportion of NVR can be displayed and recorded with auxiliary equipment.

## Automated Final Cleaning, Rinsing, and Drying Station

A typical automated final cleaning, rinsing, and drying station (Figure 3) includes an in-line filter holder/counter and an in-line nonvolatile residue sampler/evaporator installed in a laminar flow bench. These devices are plumbed to the fluid lines of a commercial, automatic final cleaning, rinsing, and drying machine.

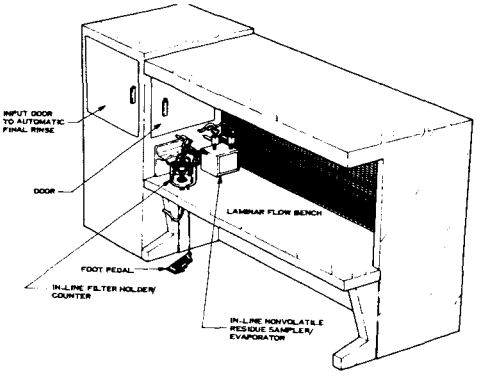



Figure 3. Automated final cleaning, rinsing and drying station

The characteristics of this machine are

1. An enclosed chamber has doors for access both to an uncontrolled environment and to a controlled environment adjacent to the machine. If the machine is used in a controlled environment rather than connected to one as shown in Figure 3, only one door is necessary. The controlled environment is a commercial laminar flow bench abutted to the final rinse machine.

2. Components placed in the enclosed chamber are subjected to a preprogrammed sequence of operations. These operations consist of forceful impingement of solvents or solutions, ultrasonic or sonic energy applied to the components while immersed in some energy-transmission liquid (which may also be a solvent or solution streams impinging on the components), flushing by chamber fill and drain, clean solvent rinse by immersion or spray, vapor degreasing, drying by heat and/or evacuation, and cooling to approximately environmental conditions. The machine need not incorporate all the energy input capabilities. Only one processing fluid stream impingement operation and one sonic or ultrasonic energy input operation in the cleaning sequence are necessary. Either or both of these operations must be applied to the final rinse or rinses. The particular combination of operations is not critical to the concept if the energy inputs are preprogrammed, and controlled as to nature, intensity, and duration.

3. The chamber drains at a selected rate into a storage reservoir during the final solvent rinses. Draining is complete after the rinses.

The NVR sampler/evaporator is installed (in terms of fluid flow) on the cleaning machine in series with or parallel to the filter/counter. Some agencies consider the filterable solids to be a portion of the NVR while others stipulate that the NVR will consist only of materials soluble in the carrier fluid. The decision to remove filterable solids by the filter/counter before or after the sample stream enters the NVR sampler/evaporator governs whether the flow shall be in series with or in parallel to the analytical equipment.

If the NVR is defined as consisting only of solubles and or particles smaller than some specified size that have passed through an absolute rated filter, the filter medium will be used in the filter/counter and the NVR sampler/evaporator will be plumbed in series with the filter/counter downstream of the latter.

The automated final cleaning machine can be used with the combined filter/counter and NVR sampler/evaporator or with either item separately.

NVR determination or particle contamination determination may be separate definitive criteria parameters of some specifications, while both criteria may be applicable to others.

The automated final cleaning, rinsing and drying station operates as follows:

1. The operator places the equipment to be cleaned in the cleaning chamber of the machine and initiates the automatic sequencing of the machine.

2. The operator, as necessary, installs a clean filter medium in the filter/counter and a clean, preweighted, NVR cup in the sampler/evaporator.

3. When the cleaning and rinsing sequences are completed, the operator examines the filter surface for particle matter in accordance with the applicable contamination control criteria specification, removes the processed NVR cup from the NVR sampler/evaporator, and weighs it (or only reads the result if a completely automated NVR device is used).

4. If the determined contamination levels meet the applicable criteria, he removes the equipment from the cleaning chamber of the machine through the door opening into the clean environment. He then proceeds with the assembly and/or sealing operations pertinent to the equipment being processed.

5. If the determined levels do not meet the applicable criteria, the operation is repeated.

Depending on the number of particles permitted, per size range, by the criteria specification, it may not be necessary for the operator to replace the filter medium in the filter/counter between cleaning/evaluation cycles. He may, instead, subtract each determined count from the preceding count and qualify the cleanliness of the equipment by the difference. The same procedure may apply to the successive weighings of the NVR cup.

The automatic sampling/sample-preparation equipment that is integrated with the machine is installed in a laminar flow bench abutted to the machine. However, the bench is not an inseparable portion of the concept. The sampling assembly could, for instance, be in any environment sufficiently clean to allow qualified parts to be removed from the chamber without being recontaminated by the environment above the level permitted by the applicable criteria. This environment could be a laminar flow clean room, a conventional clean room, or simply a controlled manufacturing or processing area depending on the stringency of the applicable contamination control criteria specification. The equipment may be installed anywhere, at any distance from the machine, on any surface, providing that the evaluation-rinsing effluent stream is delivered to it at a velocity sufficient to transport the largest and heaviest particles of interest to the applicable contamination control criteria specification.

The type of commercially available cleaning machine shown in Figure 3 is the upright, front and side loading, cabinet type. However, any configuration that will provide the minimum capabilities may be used.

A separate vacuum pump for the NVR sampler/evaporator or for the filter/counter may be provided, or the vacuum capability already existent in a commercial machine may be used. The only requirement is that the vacuum pump be protected from solvent fumes by scrubbers and absorbers.

## Summary

Although development of automated final cleaning, rinsing, and drying has progressed, no one has taken the very logical step of making these operations the parameters of particle contamination evaluation and rinsing. It is not a well understood fact (outside of contamination-control circles) that the sole quantitative content of all vehicle fluid system cleanliness criteria specifications for components lies in the content of the final rinse, the parameters for which have never been quantitatively defined. All effort expended on this activity in the vehicle aerospace field has relied on criteria that have not been dimensionally defined by determinative parameters, at least with regard to force of impingement, velocity (or "volume" of flow), orientation of surface being sampled, accessory sampling energy (e.g., insonation), or other characteristics that vitally affect the validity of results. The effort has depended almost totally on operator interpretation. As a result, reproducibility of final rinse results (and hence specification conformance) has been very unsatisfactory from program to program and, in some cases from component to component. The method described in this paper should provide pushbutton reproducibility for the entire space vehicle industry, thus permitting interprogram interchangeability of cleaned components. The method would also offer significant savings in personnel time and handling costs, and increased reliability. Finally, the method would permit the use of more stringent particle level criteria.

# 5. A UNIFIED CLEANING FACILITY IN A CLASS 100 CLEAN ROOM

by

J. A. KENAGY

Specialties Department
Sandia Corporation
Albuquerque, New Mexico

## ABSTRACT

A downdraft clean room has been equipped for cleaning and potting in a development shop which provides support to various laboratory operations. This particular clean room was designed and built to comply with Federal Standard #209 for a Class 100 room. Mechanical and electrical components are cleaned and potted in this clean room and the necessary auxiliary equipment has been installed in the room for processing a variety of plastics systems. Special care has been given to handling toxic and flammable materials. An ultrasonic cleaner is used for rough cleaning and a final rinse station is used for final cleaning. An indium surface cleanliness tester is used for checking components before potting is done. Semiautomatic vacuum chambers deaerate resin systems during the processing operations. Curing ovens and an autoclave are also used for post cures on various plastics systems.

The clean room facility in operation in the Development Shops at Sandia is designed as a portable unit to permit reasonable portability from one building to another. The room is fabricated in five sections and bolted together. Auxiliary equipment for cleaning mechanical molds and components and for processing various plastics compounds is installed along the walls.

The clean room uses downdraft laminar flow and is designed and fabricated to comply with Federal Standard #209 for a Class 100 room. Tests after installation showed the dust count to be zero for particles of 0.5 micron and a larger count per cubic foot. The room is 10' x 25' and was built to Sandia specifications by Envirco Inc. of Albuquerque. The room is located in the Plastics Shop inside the technical area. The molds, as well as parts being potted, must be cleaned and mold released, assembled, and preheated before the potting operation.

Some of the plastics systems must be preheated and deaerated before mixing and evacuated after mixing. Several of the plastics systems require pouring into molds while they are under a vacuum of 1 to 3 torr and with additional heat to maintain the proper temperature. Potting and casting operations are performed with a variety of plastics systems and hardners or catalysts. These include epoxy systems cured with agents such as benzyl dimethylamine, and other amines. The hardners present the major problem since they have a greater irritating effect on the skin than the resins. Polyester resin systems are used, and these systems are usually catalyzed with one of the peroxides, such as benzyol peroxide.

Since these materials are also flammable, certain additional precautions must be taken in handling them in a clean room. Urethane resin systems are also used, but most of these are the pre-polymer systems in which the diisocyanate and the other components have partly reacted before the manufacturer ships the system. The diisocyanates are toxic and irritating. Polyurethane foam systems are also used in a variety of applications. The fumes emitted during the foaming reactions are quite toxic, and the mixing and foaming operations are performed on one of the downdraft benches located inside the clean room.

The Industrial Hygiene Division was called in to review operations and to recommend precautionary and emergency measures regarding the handling of the toxic and flammable materials. Suggested maximum quantities and dissipation times for certain toxic and flammable materials were spelled out by members of this organization. The essence of their recommendations was that minimal quantities of amine hardners, acetone and other flammable or toxic materials, should be taken into the clean room.

Figure 1 shows a cross-sectional sketch of the clean room and part of the auxiliary equipment. The first blower (left) supplies makeup air to the clean room. Before air is introduced into the clean room it is temperature and humidity regulated. Note that the discharge from this blower is upstream and below the floor grating. This provides better mixing of the preconditioned makeup air. The next blower has a bank of five blowers that recirculates the air. The discharge from these blowers enters the plenum chamber and forces air down through the filter bank located in the ceiling. The major portion of the air passes through the floor grill and into the suction side of the five blowers for recirculation. The next blower is connected to

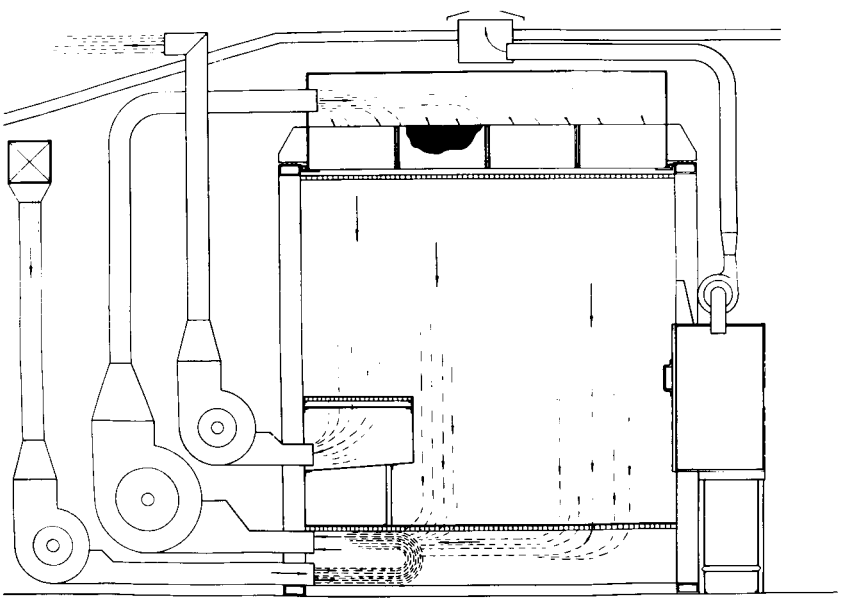


Figure 1. Cross-sectional sketch of clean room

three downdraft benches located along the north wall of the clean room. The downdraft air which enters the benches is discharged through this blower and out through the roof. Weighing, mixing, and pouring of toxic materials are performed on one of these three benches. Dampers are located in each bench to permit re-balancing of the air flow as equipment is placed on a bench.

Three plastics curing ovens are located on the right-hand side of the clean room. These ovens are face mounted in the clean room and each oven is equipped with its own exhaust blower, which is also vented to the outside through the roof. The ovens are standard commercial items; they are stainless steel lined with cross-convection re-circulating hot air. Each oven is equipped with cam-operated con-troller recorded instruments.

Figure 2 also shows a cross section of the clean room and ad-ditional auxiliary equipment. The apparatus on the left is a Westing-house re-circulating ultrasonic cleaner and final rinse station used in cleaning piece parts before assembly or potting operations. Parts being cleaned are lowered into the area and sprayed with a hand-held adjustable nozzle sprav gun. An autoclave is face mounted along the right wall. The small autoclave is capable of pressures up to 250 psi and temperatures up to 400°F. The unit is used for curing plastics systems which are thermal setting and which require densification dur-ing the curing cycle. The pressurizing media is nitrogen. Provisions have been made for operating the vessel under vacuum for bakeout or for plastics systems requiring vacuum applications during the cure cycle.

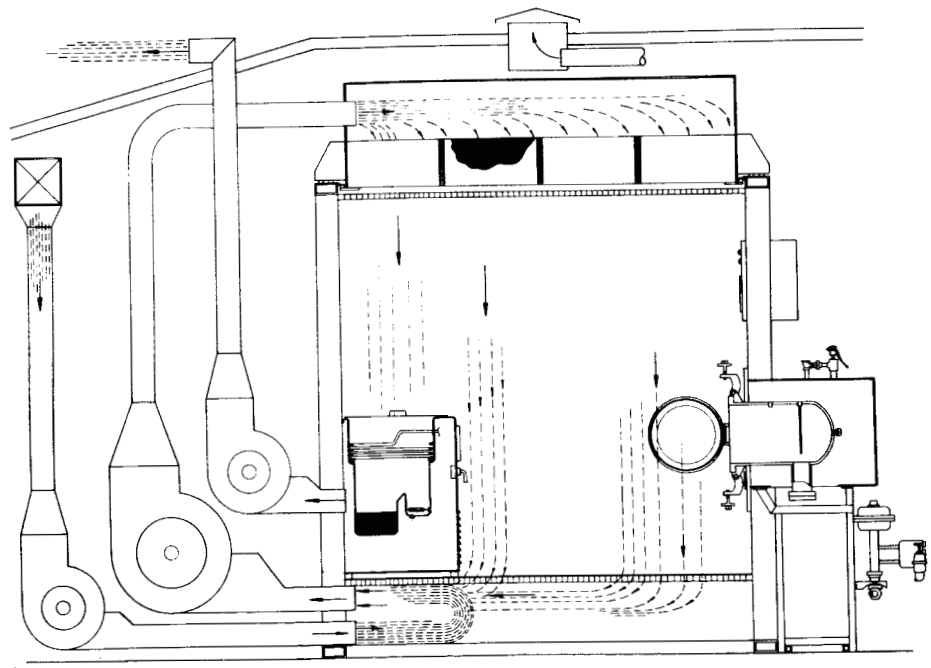


Figure 2. Another cross-sectional view of clean room

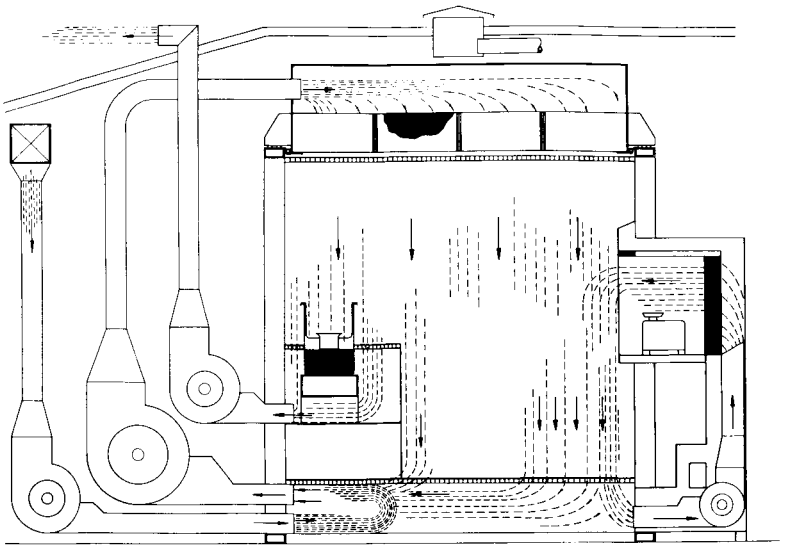


Figure 3. Cross-sectional view showing ultrasonic cleaner

Figure 3 is a cross section of the clean room showing a conventional ultrasonic cleaner mounted in one of the downdraft benches. This unit, manufactured by Turco has a tank of about 14 x 24 inches to provide space for various combinations of beakers of different cleaning and rinsing liquids. A cross-flow clean bench has been installed in the right-hand side of the clean room. This bench is complete with its own blower system for recirculating the air and filter bank located at the rear of the bench. Delicate weighing operations are performed in this clean bench, and the cross-flow permits more accurate weighing than downdraft out in the main clean room. Cleanliness tests are also performed in this bench with an indium surface cleanliness tester. Special consideration was given in the design and installation of this clean bench to isolate mechanical vibration. This is necessary for successful operation of the indium cleanliness tester.

Figure 4 shows one of two automatic encapsulating machines designed by Mr. Joff Myers, a mechanical engineer in the Development Shops organization. These units were also fabricated in the Development Shops. They are used for evacuating resin systems and also have the capability to maintain temperature during the processing cycle. Provisions have been made to introduce resin systems while components are under vacuum. These machines are used for potting and impregnating operations.

Figures 5 through 8 show closeups of the equipment. These are, respectively, (1) a front view of one of the downdraft clean benches; (2) the ultrasonic cleaning tank mounted in one of the downdraft benches; (3) the final rinse station and the ultrasonic cleaner mounted in the clean bench; and (4) the final rinse station in operation.

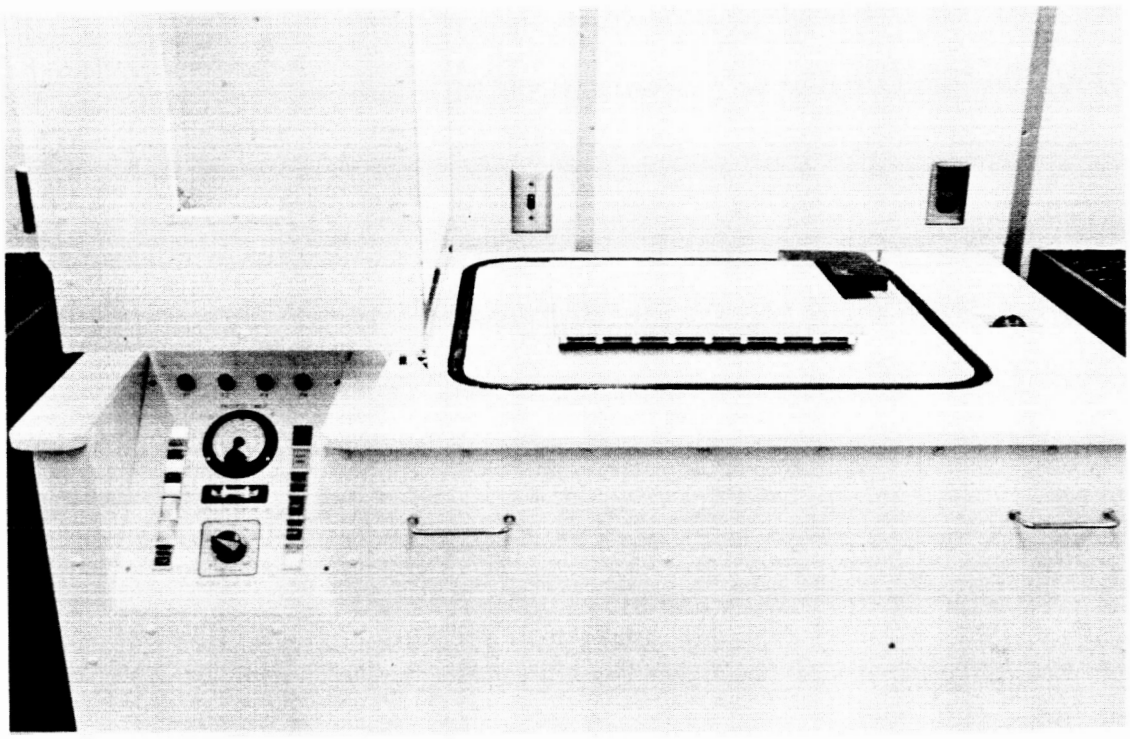


Figure 4. Automatic encapsulating machine

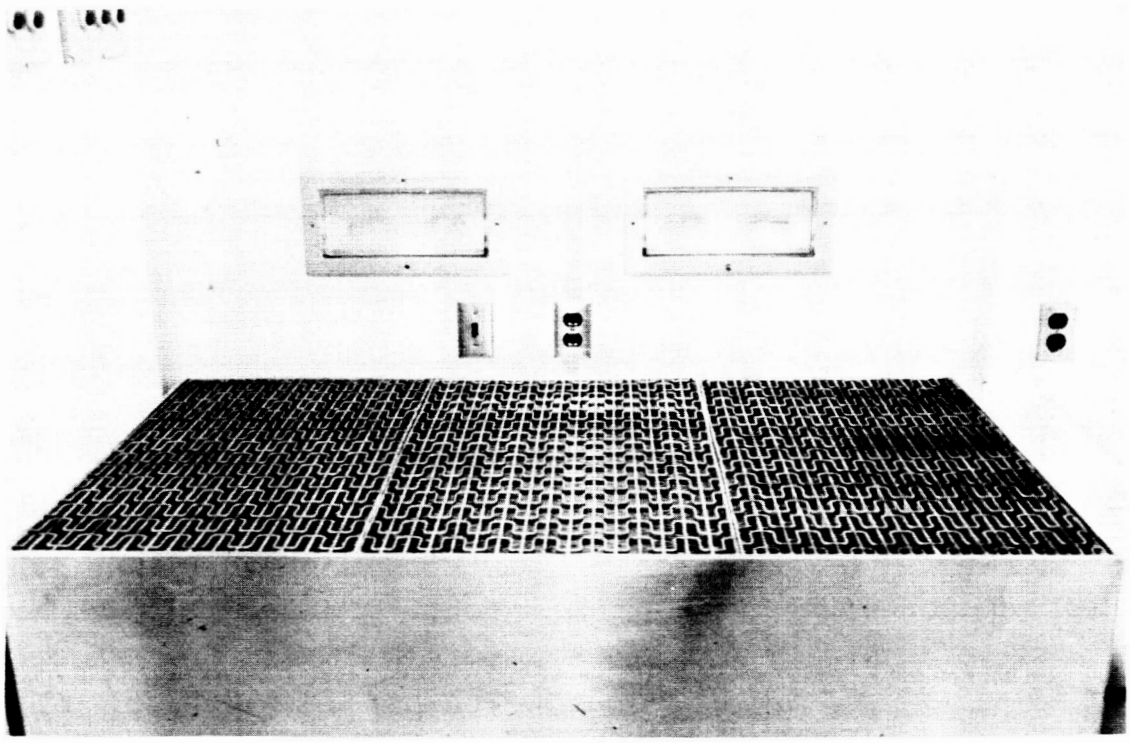


Figure 5. Front view of downdraft clean bench

Figure 6. Ultrasonic cleaning tank


Figure 7. Final rinse station (left) and ultrasonic cleaner mounted in bench

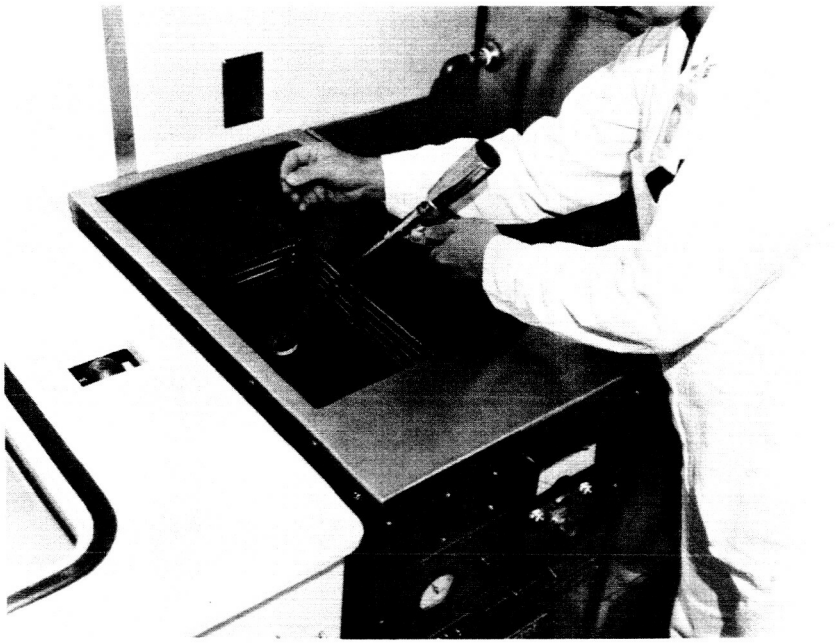


Figure 8. Final rinse station in operation

Figure 9 shows the cross-flow clean bench. The Metler scale on the right has a capacity of 800 grams. It is a direct reading scale with an accuracy of 1/10th of a gram. The indium surface cleanliness tester is located on the left. The control panel is situated on a shelf above the other equipment. The small heated pot contains molten indium; next to it is a recorder, and the next item with the large base is the indium tester.[1]

Figure 10 shows the ovens, which are face-mounted along the south wall. Since the clean room is kept under a slight positive static pressure, the air leakages through the ovens and instruments present no problem. Air leaking into the front of the ovens when the doors are opened is clean air, and all air leaks around the oven frame work are outside the clean room. There are sprinkler heads mounted along the wall above the instruments in case of fire in the ovens or in the room. There is also a pop-out escape door located in the far end of the clean room. Should a fire occur between a workman and the main entrance door, he could escape through the other end of the clean room.

Figure 11 shows the autoclave, which is also mounted along the south wall of the clean room. The face-mounted recorder controllers are for temperature and pressure control. This autoclave was manufactured by the Red Point Company, Los Angeles, California. Finally, Figure 12 gives an overall front view of the clean room.

---

[1]Papers 8 and 9 of this session deal with the indium tester.

Figure 9. Cross-flow clean bench

Figure 10. Face-mounted ovens

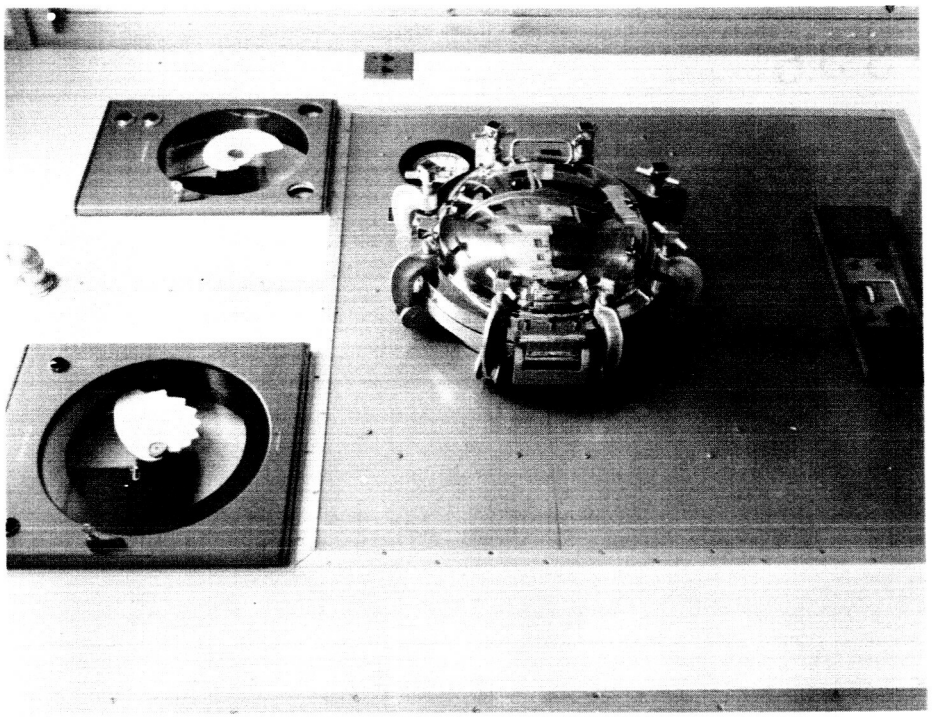


Figure 11. Clean room autoclave

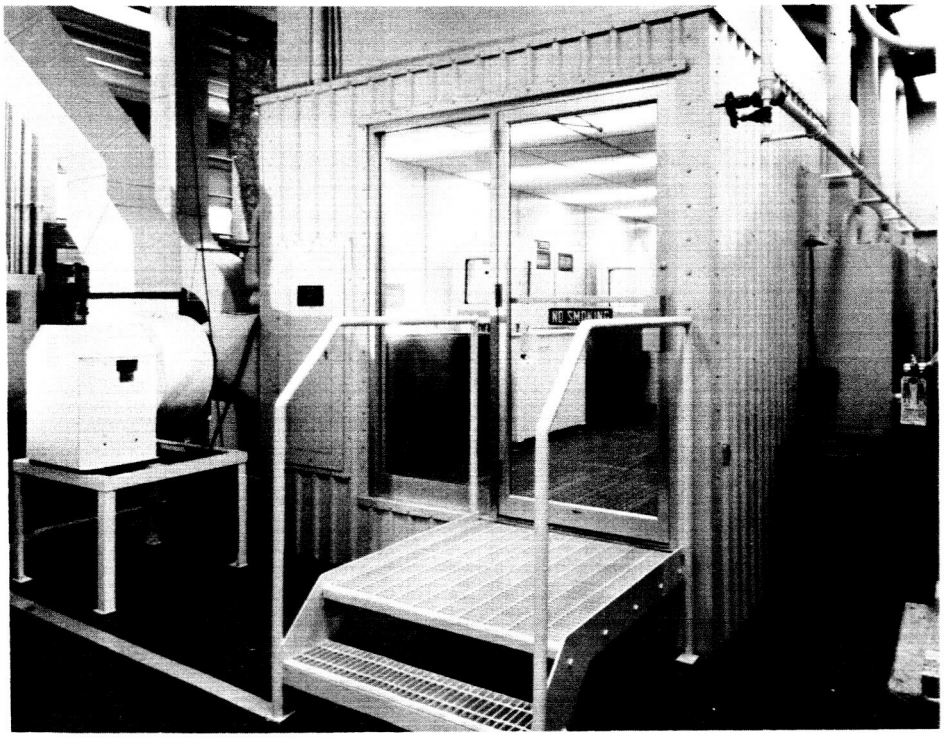


Figure 12. Overall front view of clean room

# 6. EVALUATION OF AND REQUIREMENTS FOR AUTOMATED CLEANING EQUIPMENT

by

R. B. HEDRICK

The Bendix Corporation
Kansas City, Missouri

## Introduction

At the present time, few manufacturers make standard line automated cleaning systems which include automatic handling methods and multisolution cleaning. The reason is that each user of cleaning equipment has his own cleaning process and he uses solutions, time cycles and scrubbing methods developed for the particular application. This problem is magnified when engineers within the same organization specify different cleaning processes for related products. These practices make it difficult to use automated cleaning systems.

Before automated cleaning systems can be considered, the particular cleaning process must be firm and the product schedules and cleanliness requirements of the product must justify the added expense of an automated system. Confidence in a particular cleaning process is important since the flexibility of most automated cleaning systems is somewhat limited.

## Cost

Automated cleaning systems will cost from 2 to 10 times as much as conventional cleaning equipment, which performs essentially the same function. The actual cost is proportional to the complexity, size, and number of different controls and monitoring features. The additional cost, however, can be justified if the following factors are carefully considered:

1. If part cleanliness is critical to product operation or manufacturability, the uniform results obtained through the use of automated equipment may reduce rejects, and the corresponding reduction in rework time and scrap may offset the additional cost.

2. If solvent reclamation equipment is to be made a part of the automated system, the cost savings in solvents alone is significant.

3. Automatic cleaning systems require fewer man hours than conventional cleaning systems because the operator is free to load and unload parts or perform related duties while other parts are being automatically cleaned. Solvent handling, changing, and disposal time is also sharply reduced.

## Seven Stage Automatic Cleaner

In November 1963, the Kansas City Division of the Bendix Corporation saw the need for an automated cleaning system. The need was based on the manufacture of a complex miniature electro-mechanical device which required precision cleanliness. Production schedules were sufficient to justify automated equipment. The quality requirements of this product dictated the value of uniformity in the cleaning process (such as is obtainable only through automation of the cleaning cycle).

Three basic cleaning processes were used once the parts reached the clean room assembly area (see Figure 1). These are as follows:

Cleaning Process No. 1 - A two-Stage cleaning process used to clean piece parts before assembly (see Figure 2):

Stage 1 - Ultrasonically clean for 1 to 1-1/2 minutes in trichloroethylene and blow dry.

Stage 2 - Ultrasonically clean for 2 to 5 minutes in isopropyl alcohol and blow dry.

Cleaning Process No. 2 - A four-Stage cleaning process used to clean assemblies:

Stage 1 - Ultrasonically clean for 1 to 1-1/2 minutes in trichloroethylene and blow dry.

Stage 2 - Ultrasonically clean for 2 to 5 minutes in a detergent solution and drip dry for 5 to 10 seconds. The detergent solution consists of 80 percent isopropyl alcohol and 20 percent deionized water to which is added 0.2 percent Renex 690 (a nonionic wetting agent) and 0.05 percent Span 80 (a nonionic emulsifier). The proportions given are in percent by volume.

Stage 3 - Ultrasonically clean for 2 to 5 minutes in isopropyl alcohol and blow dry.

Stage 4 - Repeat Stage 3, using fresh solvent as a final rinse.

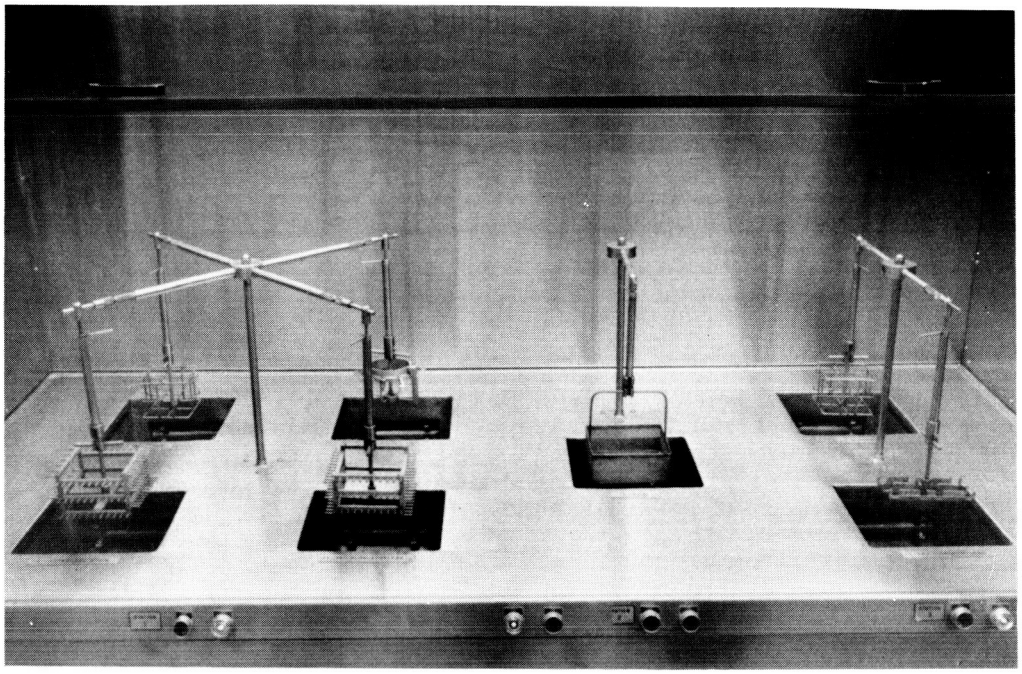


Figure 1. Seven-stage cleaning station encompassing three separate processes

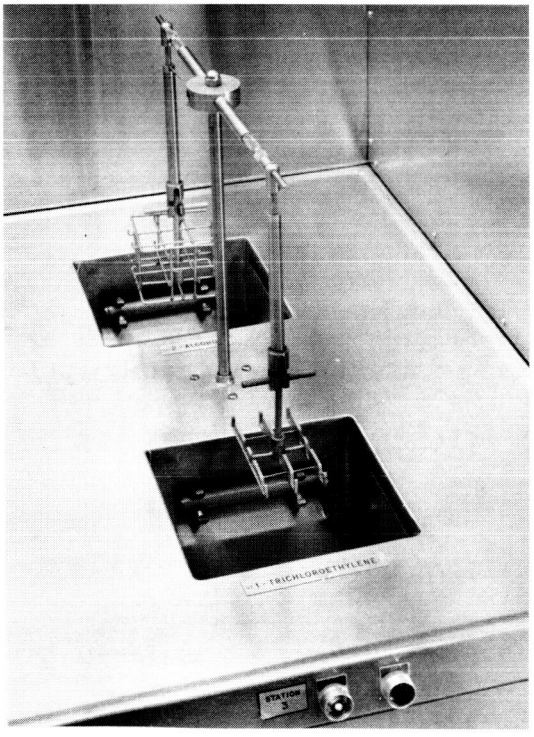



Figure 2. Two-stage cleaning station

Cleaning Process No. 3 - A one-stage cleaning process used only for those few parts that are not compatible with the solvents used in the other cleaning processes.

Stage 1 - Ultrasonically clean in trichlorotrifluoroethane (Freon PCA or Genesolv D) for 2 to 5 minutes and blow dry.

The blow dry operations each use 15 psig dry nitrogen, and each cleaning process is followed by a vacuum bake.

Based on the three cleaning processes, specifications were prepared for a custom-designed automatic cleaning cycle system. The specification defined a seven-stage cleaning unit that would provide an automated means of cleaning parts by using any one or all three of the cleaning processes described. Bendix Pioneer-Central Division designed and built the seven-stage automated cleaning unit.

## Unit Console

Because of the anticipated complexity of this type of equipment, the unit was assembled into two separate stainless steel consoles. One console contains seven ultrasonic tanks and a minimum of mechanical and electrical components necessary to operate the ultrasonic transducers and carrousel handling systems. A second console contains the ultrasonic generators, timing devices, solvent storage and filtration systems, and other mechanical and electrical components.

By housing the unit in two separate consoles it was possible to locate in the clean room only that portion of the equipment required for cleaning of the product. The bulk of the mechanisms that might require maintenance was housed in a console which is located outside the clean room (see Figure 3). Holes are cut in the wall between the two consoles to accommodate the necessary piping and electrical connections.

## Operating Sequence

The cleaning sequence in each of the seven tanks is basically the same, except that cleaning time in the two trichloroethylene tanks is shorter than in the other tanks and the parts are not blown dry following the detergent cleaning operation. Experience indicates that a white film residue will often remain on parts if they are blown dry following detergent cleaning.

The basic cleaning sequence in each tank is as follows (see Figure 4):

1. The operator places a fixture load of parts on the carrousel arm and pushes the start button. Solvent is sprayed on the walls of the tank to remove any residues remaining from a previous cleaning operation or as might be deposited between operations. The drain remains open during this step and the cleaning fixtures remain suspended above the tank at this time.

2. The drain closes and the tank is filled with solvent to a depth of 5 inches, which enters the tank through spray nozzles. An overflow drain prevents overfilling.

3. The ultrasonic energy is activated and the parts are automatically immersed into the solution to be ultrasonically cleaned for a preset time.

4. After ultrasonic cleaning, the parts are raised to a position just above the solvent level and the drain opens. A solvent spray begins and rinses the parts with clean, filtered solvent.

5. The solvent spray stops and a blow-dry cycle begins. Dry nitrogen which enters the tank through a series of spray nozzle is used to blow the parts dry. The tank drains completely during the blow-dry step.

6. The carrousel handling mechanism raises the fixture load of parts out of the tank and rotates as required to position the parts over the next-in-line cleaning tank. The machine then shuts itself off.

A lack of complete flexibility is evident in a system such as this. The time interval for each phase of the cycle is controlled by a variable timer, but the handling feature limits overall flexibility. The shorter time cycle in the trichloroethylene tanks (1 to 1-1/2 minutes) is accomplished by starting to drain the tanks at the end of 1 minute and by making sure the tank is dry after 1-1/2 minutes. Parts in the other stages of the respective cleaning process are not affected by this shorter time cycle in the trichloroethylene tank.

## Solvent System

This unit has five closed solvent systems for the seven cleaning tanks. Figure 5 illustrates the typical solvent system. Solvent is pumped from a reservoir through a rough and final filter into the cleaning tank and back into the reservoir. Redistillation systems were considered as part of a solvent regeneration system, but due to the added complexity and considerable extra cost, this feature was not included as part of the final specifications.

Each time a new cleaning cycle is started, solvent is pumped from the reservoir through a rough and final filter before reaching the respective cleaning tank. The rough filter is a depth-type cartridge unit designed to remove particles larger than 5 microns. The final filter is a membrane-type (142 mm size) originally designed to remove all particles larger than 0.5 micron.

Several problems associated with the solvent systems required correction. The original equipment had a gravity drain used to move solvent from the cleaning tank to the reservoir. This type of drain did not move solvent rapidly enough, so an air-powered solvent return pump was added to each system. The 3-gallon reservoirs provided were of insufficient capacity to support continuous operations because a small amount of solvent is lost through the exhaust during each cycle. Auxiliary reservoir tanks were added to the two solvent systems originally designed to handle two cleaning tanks. Plans have been made to add an automatic-fill system on each reservoir to maintain the required operating level.

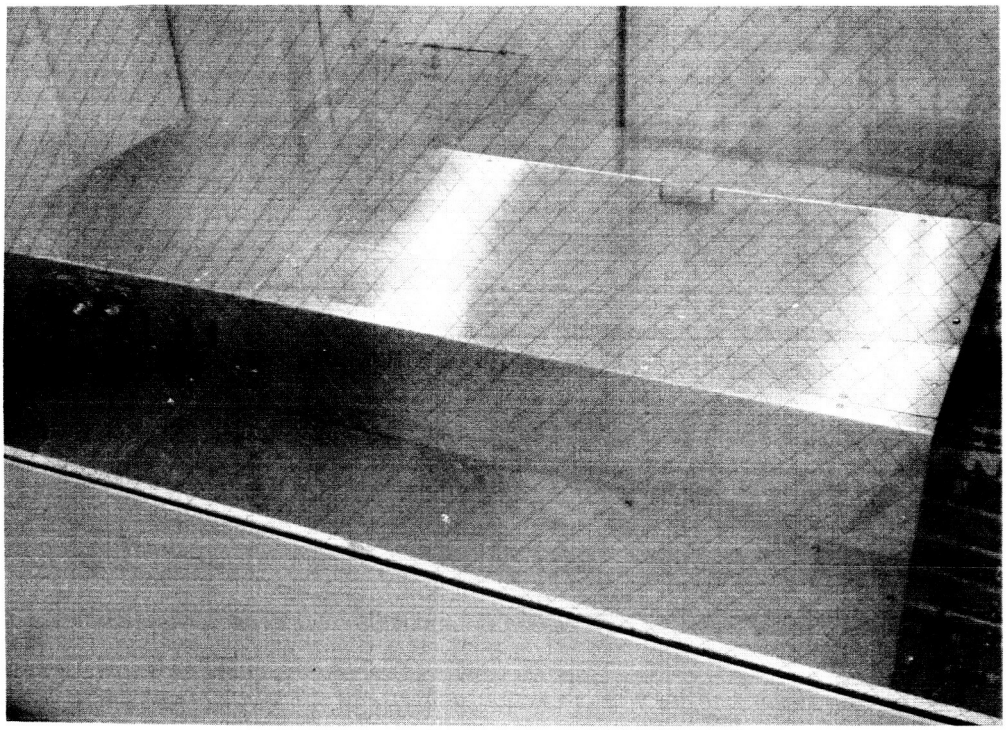


Figure 3. Operating console (outside clean room)

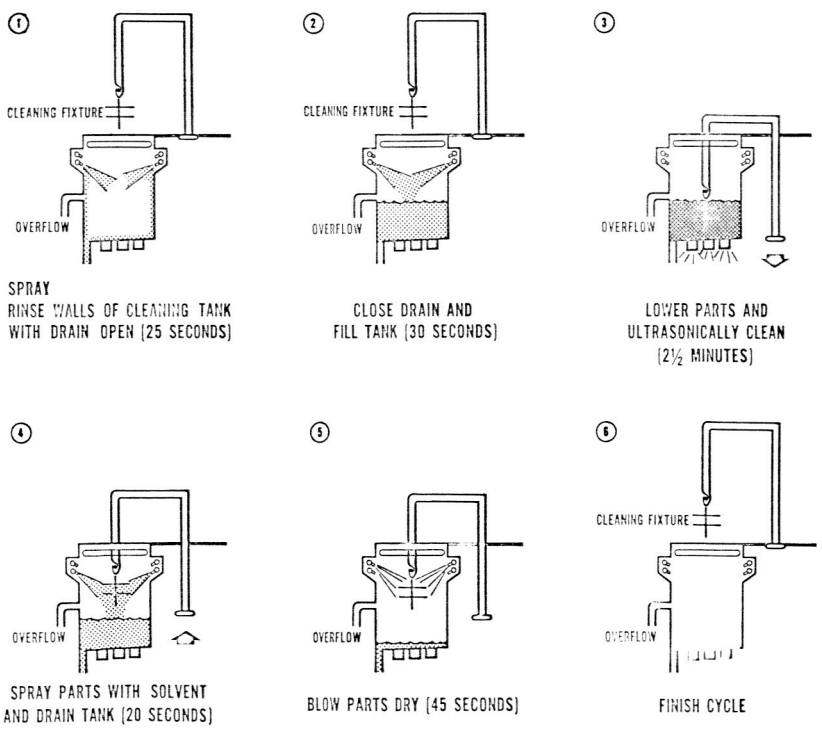



Figure 4. Automatic cleaning cycle

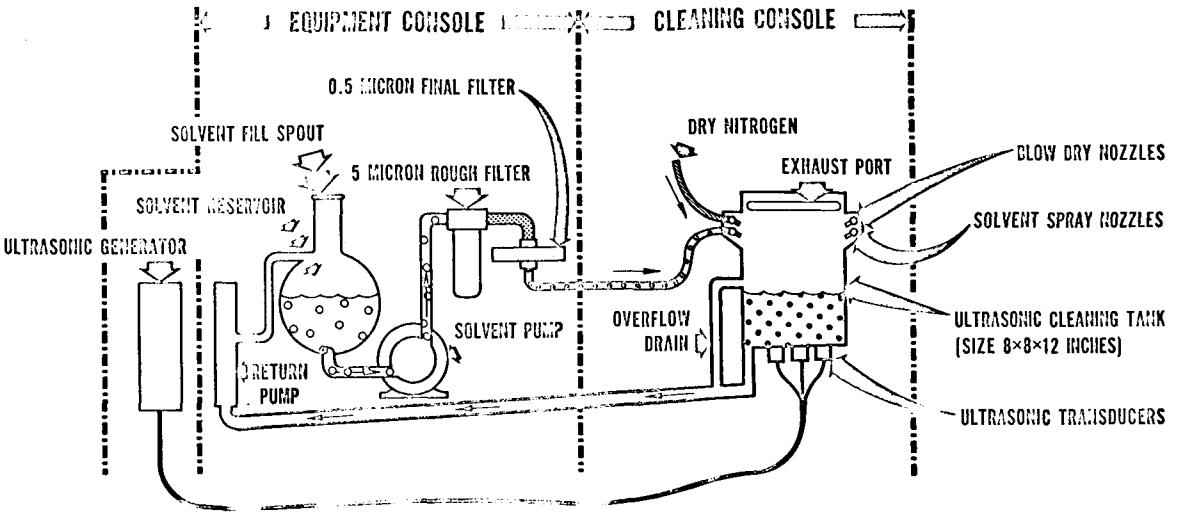



Figure 5. Individual tank schematic

When solvent quality was evaluated by counting particles and checking for dissolved contaminants (nonvolatile residues, (NVR), it was discovered that the rough filters themselves added dissolved contamination to the solvents. At this time, Bendix has not found a suitable depth-type filter which does not give off NVR, although several brands have been tested. To minimize NVR contamination, the rough filters are now washed before installation. Since the rough filters do not require frequent changing (only about once per month) NVR contamination has not proved to be a major problem, although it is hoped that a suitable filter will be found soon.

The membrane filter originally used did not give off significant dissolved contamination, but the flow rate was inadequate for the three systems which use isopropyl alcohol. One membrane filter was found to have an adequate flow rate, but it too added dissolved contamination. To eliminate the addition of dissolved contamination, the filtering capability was compromised at 0.8 micron, and a Gelman, Triacetate Metricel Filter No. GA-4 is now used.

In this automated system, the solvents are refiltered before each usage but no built-in controls (such as redistillation systems) were provided to keep the solvent from being degraded by dissolved contamination. Before the machine was purchased, it was decided to institute a bulk or mass precleaning process before moving all parts into the clean room. This proved to be an important step in preventing rapid build up of dissolved contamination in the closed solvent systems. The amount of NVR accumulated in the solvent is monitored at regular intervals with a solvent purity meter developed by Sandia Corporation. The solvent is changed when the NVR content exceeds 75 ppm (parts-per-million). Under conditions of constant use, the solvent and membrane final filter are changed once each shift.

## Special Features

Any number of special features can be added to the equipment to simplify the operation and help ensure high quality performance. The following is a list of special features included on this equipment:

1. Red-Green (go-no-go) lights indicate when the solvent level in the reservoir is sufficient for proper operation. Red-Green lights also indicate when filter changes are required as signified by an excessive pressure drop across the filters.

2. The cleaning cabinet is built into a downward, laminar flow, clean air shower.

3. Each of the seven tanks and both cabinet consoles are vented to protect against toxic or flammable fumes. When designing an exhaust system, one should be sure to consider that some solvent vapors are heavier than air. An excessive amount of solvent can be lost through the exhaust system, which should be designed to minimize this loss.

4. All mechanisms necessary for operation of the special features are housed within the cabinet consoles. As a safety feature, the cleaning operations automatically stop if any of the service accessibility panels are opened. It has been suggested that maintenance of the equipment would have been simplified if (instead of placing the motors, pump, and electrical controls in a console) the respective items were mounted on the outside wall of the clean room.

## Benefits of the Automated Cleaning System

The seven-stage automated cleaning system has the following advantages over the conventional type ultrasonic cleaning units:

1. In the conventional cleaning units, the solvent was placed in a beaker suspended in a coupling fluid within the ultrasonic tank. The beaker type cleaning arrangement was necessary to conserve solvent and to allow for the convenient changing of solvents. The automated system automatically changes solvent and does not depend on a coupling fluid, which would introduce losses of cavitation efficiency.

2. More parts can be cleaned in a shorter time with less operator effort.

3. The unit requires less floor space than a quantity of conventional cleaners capable of the same total cleaning output.

4. By performing all cleaning operations in one unit, other equipment related to cleaning operations (such as, vacuum bake ovens and storage cabinets) can be conveniently located.

Generally speaking, this automated cleaning system has proved its value since it became operational about 2 years ago. Several new miniature assemblies now specify cleaning processes compatible with the seven-stage automatic cleaner and the future of this and other automated cleaning systems at Bendix appears promising.

## Automated Printed Circuit Board (PCB) Cleaning Equipment

Specifications have been written for a new automated cleaning unit soon to be purchased. This new system will be designed to clean copper-clad printed circuit panels before application of photosensitive resist. The new system will be considerably different from the seven-stage miniature parts cleaning unit in both design and application. Experience gained from using the seven-stage system proved valuable in preparing the specification for this new automatic cleaning equipment.

## General Automated Cleaning System Considerations

Specifications for an automated cleaning system should be built around a particular cleaning process. A logical step before writing such a specification is to contact manufacturers of related cleaning equipment and automated handling systems to determine what new and unique methods are available for consideration. The mode of cleaning may be any of the standard ultrasonic, spray, scrub, vapor degrease methods or any combination.

### Handling Systems

The handling system or method of moving parts through a cleaning sequence deserves primary consideration. The flexibility, timing cycles, size of unit, and simplicity of the cleaning operation is dependent upon the particular method selected.

The following is a list of some general types of handling mechanisms:

1. CARROUSEL - This method is ideal for systems where the cleaning time in each solution is the same. The carrousel raises, lowers, and rotates as required to position parts for cleaning and to move parts from tank to tank.

2. CONTINUOUS CHAIN - The parts move continuously following the path of the chain. This method is effective and can allow for different time intervals in the various cleaning solutions. Cleaning tank sizes are usually coordinated with the handling system to achieve the desired time cycle. Cleaning time in the various tanks can be varied by changing the speed of the chain, but this means that the cleaning time in all tanks is proportionately changed.

3. CONVEYOR TYPE - Numerous versions of conveyors are used, especially on spray cleaning systems.

4. MECHANICAL LIFT - This method raises and lowers parts into a tank and moves to the next tank on some sort of track. Many different versions are available.

Solvent Systems

The solvent system should be carefully evaluated because the ability of the unit to clean parts is dependent upon the solvent cleanliness, and the cost of high purity solvents is a major part of the cost of cleaning parts. The complexity of the solvent system is dependent on the kind and volumes of solvents required.

If the parts cleanliness is not overly critical and high volume cleaning is desired, the only solvent system required can be within the cleaning tanks themselves. By maintaining a volume of solvent in the tank considerably greater (100 to 1 minimum) than the volume of the parts being cleaned, the solvent is not rapidly contaminated. A pump and filter arrangement can be included to recirculate and filter the solvent within the tank. The solvent, however, should not be recirculated while the ultrasonic cleaning cycle is in operation.

Where parts cleanliness is very critical, it is wise to refilter the solution each time it is used. This type of solvent system is the kind used on the seven-stage automated system. In order to change and filter the solvent rapidly, a reservoir is required. Filters can effectively remove particles from the solution, but some dissolved contamination will be added during each cycle. To prevent rapid contamination of the solvent by dissolved contaminates, the volume of solvent in the reservoir should be a minimum of 10 gallons for most systems designed to clean small parts.

The filters and pumps must be carefully selected. Advertising statements that a certain filter is compatible for use with a certain solvent does not always guarantee it will not add dissolved contamination to the solvent or that a realistic volume of solvent will pass through the filter at reasonable pumping pressures. Pumps with an adequate capacity should be selected to allow for pressure drops across the filters. In addition the viscosity and weight of the various solvents are different enough to make a difference in pump capacity requirements in different solvent systems.

The more stringent solvent system would include a filtering and distillation system to regenerate the solvent each time it is used, or continuously as it is being used. With some solvents, distillation is fairly simple and not very costly, but with other solvents it is quite costly and not very practical. The greater cost of high purity solvents ($1.00 to 8.00 per gallon is typical) could, however, in many cases warrant the addition of expensive complex distillation systems. The distillation equipment and filtering system combination will guard against the possibility of using a shipment of poor quality solvent.

## Monitoring Devices

Any automated equipment requires monitoring devices to control operation and ensure safe operation. Equipment to monitor such things as ultrasonic cavitation, solvent cleanliness, solvent temperature, filter conditions, solvent level should be considered before a specification for automated cleaning equipment is prepared.

## Summary

The automation of cleaning equipment is a big step toward meeting the ever-increasing cleanliness demands of many new products. This type of equipment should not be purchased haphazardly. Careful planning is necessary to make the most of such equipment. The preparation of a thorough specification and the unit design and fabrication steps are time consuming and at least a year should be allowed in the planning to accomplish these steps.

Because the flexibility of automated cleaning equipment is limited, the equipment should be designed around a proven cleaning process. The particular cleaning process selected will have to be based on the type of parts, materials, and cleanliness requirements of the particular parts involved.

To offset the added cost of such automated equipment, the possibilities for cost saving features should be evaluated. Reclamation of solvents, labor saving features, and improved quality are obvious possibilities that can lead to ultimate cost savings.

Tests (using monitoring instruments such as liquid-borne particle counters and solvent purity meters) should be performed to check out the compatibility of materials, filters, pumps, and other equipment to be used with the respective solutions.

# 7. REVIEW OF SURFACE CLEANLINESS TESTS

by L. K. Jones

Presented by

C. W. JENNINGS

Sandia Corporation
Albuquerque, New Mexico

After reading the title of this talk, you may have been prepared to listen to what could be described as a rerun of a summer replacement TV program. Certainly, a detailed discussion of a "review of surface cleanliness tests" would be a "rerun" of what many of you have already heard at other conferences on contamination control or will hear at this symposium.

Such a review would probably include contact angle, atomizer spray, solvent extract, indium adhesion, radioactive tracer, electrical conductivity of water rinse, particle counting, gravimetric, light scattering, test cultures, and even such a common test as visual examination.

Many fine conference papers and journal articles have been prepared on the subject of monitoring for particulate, film, and microbial contamination. Mr. William Hume of McDonnell Aircraft, for one, has reviewed a number of tests in Electronic Components in the Clean Room presented at the National Electronic Packaging and Production Conference in Long Beach earlier this year. In fact, a number of excellent papers now being presented at this particular AEC-NASA Symposium describe in precise detail either specific surface cleanliness tests or specific monitoring procedures which relate to the cleanliness of surfaces. Since these papers, in a sense, constitute a review, we will discuss the problem with surface cleanliness tests and their capabilities and limitations.

Many types of tests available, specific tests, in general, are capable of detecting contaminants within a given category, that is, soluble, insoluble, organic, inorganic, film, particulate, and microbial. The sensitivity of the tests may vary from relatively insensitive to very sensitive. For example, yesterday, there were discussions on the solvent purity meter and the nephelometer technique, which are capable of detecting down to a few ppm. We heard about the high sensitivity of the liquid conductivity test. Then there is the radiochemical detection of contamination which we found could detect to fractional micrograms/$cm^2$. If proper calibration can be made with suspected contaminants, monolayer like amounts can be detected by infrared multireflectance or by the indium adhesion tester.

Each test has its limit of applicability - there is no such thing as a universal or ideal test, though we are constantly seeking, to approach such. If we had one, it would be "a simple test with a minimum of human factor involved, one which was repeatable and yielded some number on a "contamination scale" - one capable of identifying all types of contamination (organic or inorganic) on all types of substrates while being nondestructive to the substrate."

Although we do not have such a universal test, we do have a variety of tests at hand and when these are properly used in a comprehensive sequence, one can test an item for almost any combination of contaminants. In addition we have all the modern analytical chemical instrumentation techniques available to assist these tests.

If all these tests and techniques are available, what then is the problem?

The problem I think relates largely to personnel.

1. We need the depth to be knowledgeable of the chemistry and mechanics of contamination - prevention, removal, and detection -- and the breadth to be knowledgeable of the many situations where contamination may arise.

2. We need to have the capability of integrating this knowledge with the varying conditions of production and final use of the various items.

3. We presumably recognize the problems of contamination prevention, removal and detection; we need to have others recognize these problems and to implement what is known in actual production.

While a very talented and versatile person could fill all the needs listed, we would find that, because of the scope of the problems, a selected group with varying overlapping talents would be required to provide the depth, breadth, and capabilities that are necessary. Meaningful cleanliness tests can be applied to production items only through the cooperative efforts of several different disciplines.

For example, the tests have to be applied to actual components or parts or structures being produced in plants, often some distance away. One must determine the relation of cleanliness to the function of the part or final assembly. How much contamination can be tolerated and how is this to be determined and monitored? Here the group with its varied experience and disciplines needs to coordinate with the design and production engineers to achieve practicable workable solutions.

In many industrial and governmental organizations, we find a control group that is usually too small, if it exists at all. This group often has little responsibility and essentially no authority for production items and is permitted to serve in its control capacity only when called upon. As a general rule, the overall effect of control is sporadic and fragmentary because of the tremendous range of the problem.

This does not detract from the efforts of these control groups, or from the studies being performed on old and new control procedures, or the program any organization may have in connection with contamination control. It is a hard fact, however, that we have been caught

up in a new requirement in technology where the supply of knowledgeable people is short and the problems are many. Someone has suggested that specialized training of the college level be provided in contamination control. We heartily agree that all haste should be made in this direction and we would recommend that considerable thought and planning be given to the organization of such training. It is also indicated that considerable thought should be given to the organization and management of the control effort.

All of this is well and good for the larger organization which can provide sufficiently trained personnel and adequate funds to handle its in-house problems. What about the smaller organization who either does not have an adequate staff or has no staff at all? Many of these organizations are entering into a highly competitive and very demanding market. They will be forced to look for the talented and versatile person mentioned earlier. In the meanwhile, it is imperative that we as AEC and NASA design agencies - and other large organizations - recognize that very close liaison is necessary if the contractor, small or otherwise, is not "up" on contamination control. We are quite prone to assume that a specification requiring cleaning, contamination control, and cleanliness verification will give us what we want. It has been our experience this is not enough. For example, in the production of sensing devices it has often been necessary for us to educate the supplier on the need and philosophy of cleaning and contamination control. By philosophy we mean the overall cleanliness effort that one does not clean or test a part and then put it in a container where it could pick up contamination from the container during storage before the next assembly.

It is possible we are belaboring the obvious and that evolution with time will slowly eliminate the problem. As more and more people are trained, as the various phases of contamination control become more closely coordinated, as each group from management on down recognizes the importance of control, then perhaps the application of cleanliness tests - the moment of truth in contamination control - will no longer be a problem.

# 8. PRINCIPLES OF OPERATION OF THE INDIUM ADHESION TESTER USED FOR SURFACE CONTAMINATION MEASUREMENTS

by

G. L. KRIEGER
Electro-Explosives Division
Sandia Corporation
Albuquerque, New Mexico

## Abstract

The basis for the use of solid-solid adhesion as a measure of surface cleanliness is discussed. Equipment and procedures are described for practical use of the effect, and typical test results are given.

The performance of the indium adhesion test for surface cleanliness is based upon the phenomenon of adhesion between solids. This has been the subject of study by a number of investigators for many years and has only recently become well understood. It has been known for a long time, however, that adhesion is very sensitive to surface contamination. Nevertheless, to our knowledge, there has not been any previous use made of this effect as a quantitative measure of surface cleanliness. The reason may be the peculiar difficulties of making such measurements under ordinary laboratory conditions.

Two solid substances placed in contact adhere if the interfacial energy is less than the sum of the two surface energies. Many solid materials have intrinsic surface properties fulfilling this requirement for adhesion, so that the formation of adhesive junctions is limited by surface cleanliness. However, observable adhesion does not occur between most solids even when they are as clean as modern laboratory room conditions permit. This absence of observed adhesion has previously been attributed even to strictly clean surfaces through an explanation arising out of the work of J. S. McFarlane, D. Tabor and F. P. Bowden.[1,2,3]

---

[1] Moore, A. J. and Tabor, D., Brit. J. Appl. Phys., 3, p. 299 (1952)

[2] McFarlane, J. S. and Tabor, D., Proc. Roy. Soc., A202, pp. 224-243 (1950)

[3] Bowden, F. P., Adhesion and Cohesion, Elsevier Pub. Co., New York, 1962, pp. 121-145

It appeared that although strong adhesive bonds are indeed formed, these bonds are usually broken by the release of elastic stresses in the material when the forming pressure is removed. This idea is supported by the fact that observable adhesion varies with the hardness of the substance used, sufficiently soft materials exhibiting strong adhesion when clean, presumably because of the predominance of plastic deformation over elastic distortion. Previous descriptions[4,5] of the indium adhesion test have, therefore, given the softness of indium as one of the properties responsible for its outstanding performance as an adhesive probe for surface contaminants. The fact that indium also benefits from forming a very thin self-protecting oxide film which is easily ruptured was indicated as of possible secondary importance.

More recent experiments by investigators using ultraclean surfaces have changed this picture considerably. K. I. Johnson and D. V. Keller, Jr.[6] report a large observable adhesion between the hard metals, molybdenum and titanium, when they are sufficiently clean. This certainly implies that the easy rupturing of a surface layer such as occurs when indium is pressed against another surface is the dominant factor making adhesion measurements possible under ordinary room air conditions. In other words, the hardness of a metal has an indirect effect upon its sensitivity to contaminants as an influence on adhesion. A practical test requires the use of a soft material which ignores the effect of room air contamination on its own surface and can therefore be a probe for the contaminants of interest on other materials. This is the basis for the use of indium adhesion as a measure of surface cleanliness.

This effect can be quantitatively defined in terms of the ratio of the tensile force for adhesive failure to the force used to form the adhesive junction. This is known as the coefficient of adhesion, $\sigma$. Figure 1 is a graphical representation of this observation as presented on a strip chart record. The proper performance of this measurement, although it is relatively simple, requires the observance of certain careful steps, the importance of which may not be immediately obvious. Special equipment has been constructed and used for this purpose. This is shown photographically in Figure 2 and functionally in Figure 3. Briefly, the technique consists of (1) preparing a clean indium surface by breaking the end from a small sample of indium supported on a glass rod, (2) pressing the surface of a test specimen against the indium tip with a known force, and (3) pulling the test specimen away from the indium tip, observing the force for adhesive failure. The sequence of operations involved in steps (2) and (3) must be carried out in such a way as to avoid spurious forces acting between the indium probe and the specimen surface. For this reason,

---

[4]Krieger, G. L. and Wilson, G. J., Materials Research and Standards, 5, pp. 341-348 (1965)

[5]Krieger, G. L. Electrochemical Society Extended Abstracts of Electronics Div., 14, pp. 253-260 (1965)

[6]Johnson, K. I. and Keller, D. V., Jr., Journal of Vacuum Science and Technology, e, pp. 115-122 (1967)

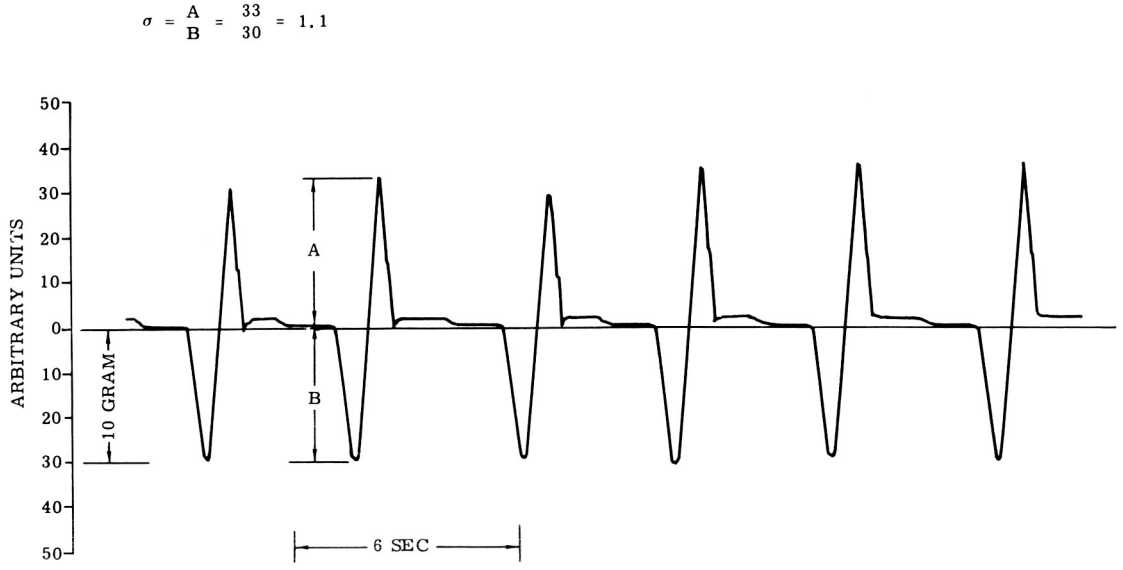



Figure 1. Fast scan of indium adhesion test pattern using clean mica

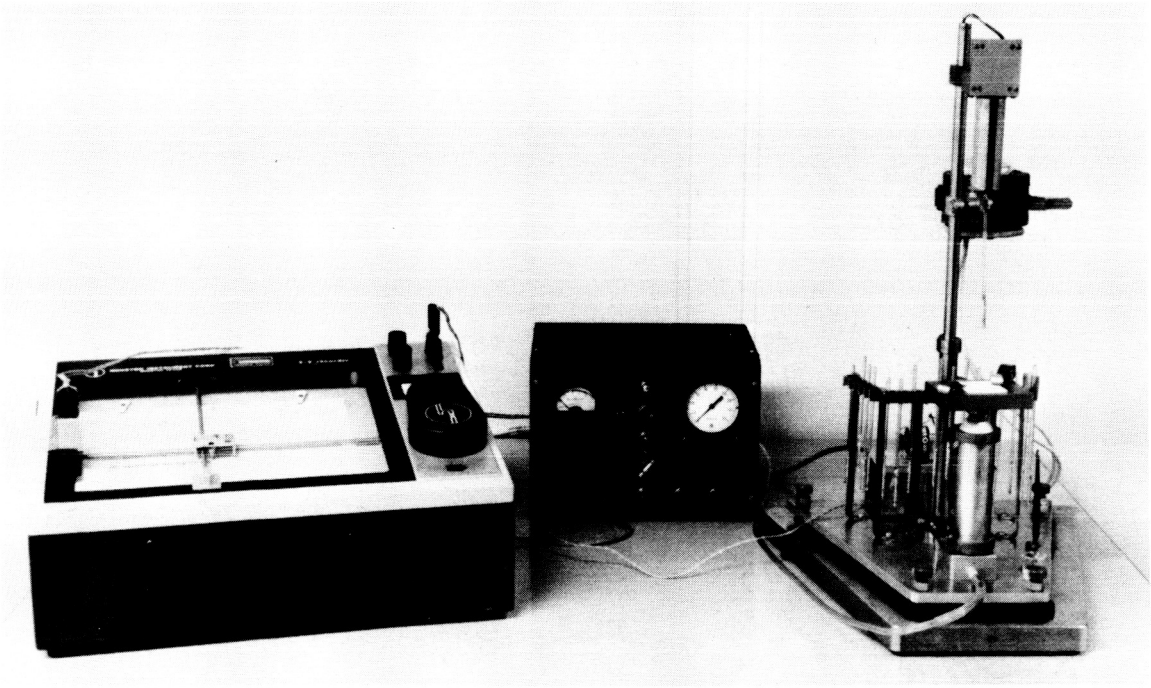


Figure 2. Indium adhesion surface tester (overall view)

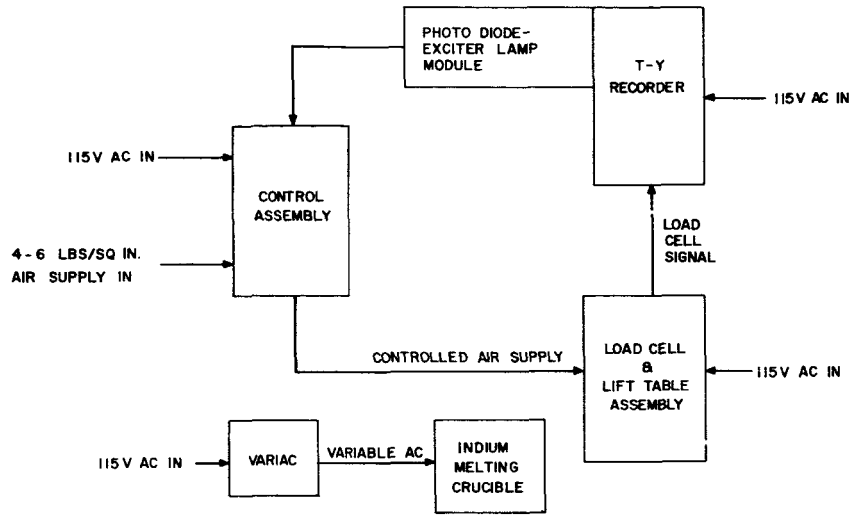



Figure 3. Simplified block diagram of IAST

the apparatus is normally provided with a vibration reducing base support. A more troublesome force that must be minimized is that of impact, which always occurs between the indium tip and specimen surface at the instant of contact. This requires keeping the rate of specimen motion less than 1 cm per second. The exact rate is not critical, but excessively slow motion makes the occurrence of accidental vibrations more likely and so is to be avoided. A rate between 1 cm per second and 0.5 cm per second has been found satisfactory with the apparatus now in use. When the test is performed with sufficient care, it yields a $\sigma$ value of about 1 on clean surfaces. The testing of a reference material such as freshly cleaved mica is recommended for confirmation of proper behavior of the equipment.

The sensitivity of the test is enhanced by taking advantage of repeated use of an indium tip when a given specimen is tested. On clean surfaces, this makes no noticeable difference because the tip is not soiled by the test. However, contaminant from even a slightly soiled surface accumulates on the tip as it is reused so that successive tests made with it will show lower $\sigma$ values. A sequence of five tests with one tip seems to be enough to indicate clearly any contamination for which the test is practically useful.

Results are recorded on a strip chart incorporated with the test equipment which automatically carries out 5 $\sigma$ measurements in sequence. The test has proven useful on specimens of greatly varying surface finishes and detects both hydrophobic and hydrophylic contaminants. Figure 4 shows typical test patterns that are observed. The results of practical application of the indium adhesion test will be given in the next paper by S. L. Smith.

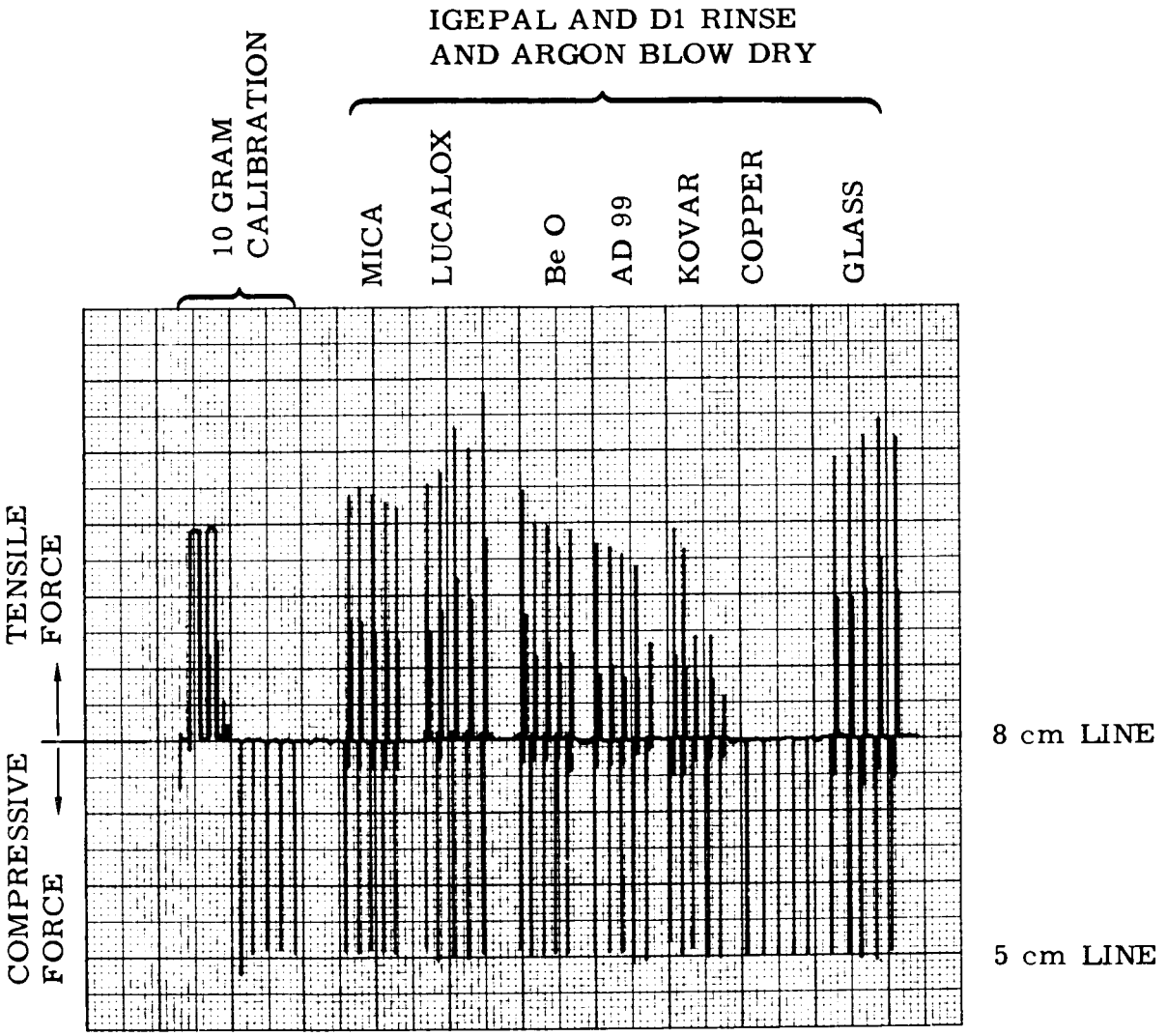



Figure 4. Typical test patterns

# 9. THE INDIUM ADHESION TEST APPLICATIONS

by

S. L. SMITH

Manufacturing Research Division
Sandia Corporation
Albuquerque, New Mexico

ABSTRACT

This paper presents a brief review of the application of the Indium Adhesion Surface Tester developed by G. L. Krieger.[1,2] Most materials used in the manufacture of electrical and mechanical devices can be tested for contamination, before and/or subsequent to cleaning to measure the improvement or level with this device. Also, there are indications that monolayer thickness measurements are within the scope of the instrument.[3]

## Introduction

During the past decade there has probably been more emphasis placed on contamination control than any other single aspect of weapon component manufacture. The primary reason for this may be that just ten years ago, systems started to become so sophisticated and dependent on cleanliness that we could no longer ignore contamination.

Following the sophistication of systems came new clean rooms, new cleaning equipment, laminar flow facilities and a multitude of processes too lengthy to mention. One of the greatest problems has been the measurement of contamination with any degree of accuracy and speed. This would provide us with data to initiate refinement and control of a process before it becomes a real problem.

## Application

The Indium Adhesion Surface Tester is as close as we have come to having a good production line audit of contamination with rapid feedback.

---

[1]Krieger, G. L., and Wilson, G. J., An Indium Adhesion Test of Surface Cleanliness, SCTM-276-63 (14), Sandia Laboratory, Albuquerque.

[2]Krieger, G. L., Improvements in Use of the Indium Adhesion Test for Surface Cleanliness, SCTM 64-1722, Sandia Laboratory, Albuquerque.

[3]Cuthrell, R. E., The Quantitative Detection of Molecular Layers with the Indium Adhesion Tester, SCDR-66-300, Sandia Laboratory, Albuquerque.

Two facilities are using the IAST on ceramic piece parts. The parts are first cleaned then tested for indium adhesion before the surface is metalized. In this case, a coefficient of adhesion of approximately 0.9 is desirable.

At Sandia, one organization uses the IAST to test substrates before the deposition of thin film circuits. Actual readings for polished silicon are between 0.8 and 0.9, for ceramic 1.1 to 1.2, and for glass 1.1 to 1.3.

Another application that was quite significant was the use of the IAST to support data in the evaluation of several ultrasonic cleaning units. The results were very good and a correlation existed with the following test methods.

- Aluminum Foil Erosion Test
- Ball Bounce Test
- Mikasonic Test
- Metal Mark Removal Test
- Goniometer (Water drop contact angle)
  Nickel and Kovar coupons were used for this and the IAST method.

The applications of the IAST unit are almost as limitless as the piece parts used in manufacturing, which require, or should require, cleaning. A few examples where the IAST could be put to good use are as follows.

- PC Boards before flow or hand soldering to detect contamination that might prevent wetting.
- Components before placement in vacuum environments to minimize evolution of unwanted gasses due to contaminants.
- Switch and relay contacts to minimize latent problems due to contamination.
- Evaluation of any type cleaning process or equipment by using sample coupons coated with known contaminants and levels and testing after cleaning for efficiency of removal of the film.
- Test for surface cleanliness on items to be electroplated.

Parts of almost any geometrical configuration can be tested. However, with the present design, size is limited and ID's and spaces too small for the indium tip cannot be checked.

For every application, the material and surface conditions must be taken into consideration because of the variation in readings from one to another. For specific uses, the coefficient values should be established for that set of conditions and the intended subsequent application of the item.

Some approximations for coefficients for various applications are as follows.

| Item | Sigma |
|---|---|
| Semiconductor Surfaces | 1.0 |
| Substrates for adherent deposit | 1.0 |
| Internal surfaces of vacuum devices | .67 |
| Surfaces for critical adhesive bonding | .67 |
| Internal surfaces of nonvacuum hermetic sealed devices | .33 |
| Noncritical adhesive bonding | .33 |

In a report by R. E. Cuthrell of Sandia, The Quantitative Detection of Molecular Layers with the Indium Adhesion Tester, his closing comments were, "The sensitivity of the instrument is such that the difference between monolayers of members of a homologous series of compounds could be detected. The linear relationship found between the coefficient of adhesion and the logarithm of the layer thickness indicates that this technique should find some application in the field of surface chemistry and in the study of wettability and adhesion in polymer arts." It may be that in addition to contamination detection, the IAST will find its way into the research lab as well.

OGLE: You mentioned detecting small amounts of oil from small bearings. How did you make the measurements?

DAVIS: By infrared spectometry, which is the method used for most of the organic contamination.

VICKERS: Mr. Saperstein, your study showing retention of trichloroethylene (tric) on aluminum surfaces suggests that this will be true also for other metallic surfaces. Preweld cleaning of stainless steels and titanium alloys often includes tric degreasing. Since these materials are highly susceptible to stress corrosion in the presence of chlorides, what do you feel is the effect of tric preweld cleaning on weld quality? Also what would be the long time effect of postweld tric cleaning?

SAPERSTEIN: It is true that tric will be absorbed on other materials as well as on aluminum and that certain materials will be damaged by its presence other than just suffering weld damage. Certainly, corrosion and stress corrosion cracking, which is surface nucleated will be influenced by contaminants that are present on the surface. However, IIT has not undertaken a study to determine the effects on certain materials. It appears that such a study would be worthwhile.

ELLENBURG: Does a citric acid chemical cleaning solution produce the same problems as the hot alkali solution?

SAPERSTEIN: We do not know. We have not worked with that solution.

YEICH: What lubricants were used to machine? If none, how was machining accomplished with spalling?

SAPERSTEIN: We used dry machining. In our work, we wanted to avoid the use of lubricants. In production, where you want long tool life and good surface finish, you have to use lubricants. I cannot say at this moment what damage would be produced by lubricants, but certainly there would be significant damage absorption of the lubricants. Most of the weld preparation on the SATURN is done dry.

BROWN: Did you use any mechanical methods, such as shot cleaning, to clean after machining?

SAPERSTEIN: We did not employ shot cleaning since we machined dry. There was no need to remove an organic residue that was produced by machining.

ELLENBURG: Is any other organic solvent less detrimental than trichloroethylene or freon to the aluminum surface?

SAPERSTEIN: This is a good question for which we have no answer at the moment. We would welcome sponsorship for a study to come up with an answer.

PEIPELMAN: Mrs. Ellenburg, what is the accuracy of determining NVR in PCA freon by using the boil down and weight method?

ELLENBURG: You would lose 30-50% of the residue, the same as for any other organic solvent by virtue of the partial pressures.

GAYLE: Does the use of the contact angle for teflon assume that this angle is equivalent to that for a typical contaminant?

ELLENBURG: Teflon was selected on the basis of surface energy considerations. A contaminated hydrocarbon surface of closely packed CH molecules will have an energy of about 36 dyne/cm. If we select either teflon, which has 18 dyne/cm, or polyethylene, which has about 24 dyne/cm, it means that a material that was spread on either of these two will have an angle low enough to be used on hydrocarbon contamination.

GAYLE: In using this angle method for evaluating cleaning solutions, did you take into account the sometimes marked dependance of $\emptyset$ on small temperature changes?

ELLENBURG: In the solutions that we used, there were some changes, and below 80°F, there were marked changes.

SMITH, V. I.: Mr. McDonald, your proposed test procedure on each batch of solvent from the final rinse assumes that the results of this test indicate the part is clean or contaminated. Is this your assumption? If so, what methods are used to test the surfaces to prove they were clean. In our experience with water clean stations, a test on the final rinse has only been an indication that a standard method has been followed, but separate tests on surface quality are recommended.

MCDONALD: Most of our test methods are on an indirect nature, and, on this basis, there is the assumption that the parts are clean. There is some visual inspection. For example, one series of parts required 100X and a visual inspection by microscope. We do not know of anyone else who is using a direct surface method. Also, some components of interest, such as valves and regulators, have internal surfaces that are hard to inspect other than by indirect means.

GAYLE: Most, if not all, batch sampling devices require purging to remove the initial slugs of trapped or generated contaminant. How do you avoid this with the in-line microscope?

MCDONALD: Before making the particulate count, we take a measure of the solvent, pass it through the microscope, and then take a background count. Then the measured sample is run through the microscope and the count is taken. If the background count is insignificant and has been so time after time, this procedure possibly could be eliminated.

MORRISON: Are residues of indium left on the tested material?

KRIEGER: Yes, in those cases where the tested material is sufficiently clean and good adhesion occurs. We have found residues on the order of 10 micrograms. Sometimes it is possible to use an area of the part on which such a residue is not objectionable. If this is impossible, then the test must be considered destructive.

GAYLE: Does the cross section area and the planarity of the tip have no effect on the sensitivity of the test?

KRIEGER: A lot of the fundamental theory was gathered from the reading of the references given in the presentation. The cross section area does not have an effect because it adjusts itself in proportion to the force that is used. This becomes Young's modulus, 1 kilogram per square kilometer. An effect would be encountered only if varying forces were used, but we presupposed a constant force. The planarity between

the surfaces would have an effect if the test surface approached the probe at a sufficient angle that a shear force would be introduced. The specifications for the test, which were not presented, include a statement that the surface must be perpendicular to the probe ±5°.

SAPERSTEIN: Elements such as indium and gallium promote stress corrosion cracking in certain metals. Consequently, what other analogous approaches with different probe materials could you suggest?

KRIEGER: We do not know of any other probe material whose properties sufficiently approach those of indium to be useful in this test. The unusual softness of the metal and its characteristic of forming a thin, self-protective oxide which is ruptured with the forces used in this test is, to our knowledge, an unique property.

SAPERSTEIN: The surface reaction between indium and substrate will depend upon substrate materials. Bond strength will depend upon this reaction independent of surface cleanliness per se. Therefore, how can the coefficient of adhesion be considered as a measure of cleanliness along?

KRIEGER: We are working in a region of adhesive phenomena where those reactions between the indium probe and the substrate depend upon the characteristics of the substrate materials. These reactions occur in the range of contaminant levels for which the test is sensitive. Clean materials are in a range somewhere below the monomolecular layer of contamination level. They would follow the responses that are suggested here; they then would require, in some cases, activation energies which may or may not be present, depending upon the requirements of this particular reaction. In any event, we cannot get an apparent coefficient of adhesion of greater than unity if the test is properly performed. In other words, the force required to break the bond will not be any greater than the force required to form it since we plastically deform the indium in both directions in the same way. Once it starts to break, it will neck down at the junction or near to it, and the force required to break it cannot get any greater than the force used to distort the indium at the time it was formed.

PEIPELMAN: Does the indium test leave enough indium to degrade the cleanliness?

SMITH S. L.: As Mr. Krieger said, the indium residue is about 10 micrograms. If it degrades the cleanliness one is looking for, then the answer to your question is "yes."

YEICH: What type of instrumentation was used to check particle count?

KENAGY: A microscope.

YEICH: What is air velocity across medlar scale in horizontal bench? Does this air flow affect the scale?

KENAGY: The velocity is about 50 feet per minute, which is about one-half of the clean room down draft velocity. The flow does affect the scale. We are working with plastics and are not dealing in micrograms. It is sufficient for us to have 1/10 gram, and our scale is not off that much. By moving it into the cross flow, we did notice a difference from having it in the downstream flow.

PEIPELMAN: Is indium compatible with various fuels and oxidizers?

SMITH S. L.: If the assumption is made that a surface is nearly perfectly clean, such as freshly cleaved mica or flamed platinum, a deposit of approximately ten micrograms of indium may be made. In this case, the amount is so small that it would be difficult to detect its presence except by flaming the surface and observing the discoloration. The amount of heat liberated as a result of oxidation of ten micrograms of indium could probably not be detected. If another assumption is made that the surface is less than perfectly clean, such as the case would be during most tests, the amount deposited would of course be much less than ten micrograms.

SESSION IV

CONTAMINATION IN AIR AND GASES

Presentations



Session Chairman


H. GILBERT
Atomic Energy Commission
Washington, D. C.

SESSION IV

# CONTAMINATION IN AIR AND GASES

## INTRODUCTION

by

H. GILBERT

Operational Safety Division
Atomic Energy Commission
Washington, D. C.

The present session entertains one of the more interesting subjects, at least to me, of the whole symposium. It is an area where everyone knows a little and nobody knows enough. It is a little difficult at this point to visualize any technology today requiring a clean environment where air or gas cleaning in some form may not be found. Historically, air and gas cleaning underwent a flurry of activity in World War II. It seemed to lapse into relative inactivity after that, and about five to ten years ago, it got a shot in the arm, and then it got further stimulation when the clean room concept came into being. This phase of the work also applies to the instrumentation and the techniques for measuring the effectiveness of air and gas cleaning, although much less effort has been devoted to the instrumentation and the techniques, which are the theme of this particular session. With the trend throughout the past and currently, anyone with a minimum of familiarity in this area will readily recognize that there is still a crying need for more development and more progress in this area. This is one of the reasons we are looking forward to the papers in this session to give us another step forward in trying to overcome this void.

# 1. CLEAN ROOM MONITORING IN THE MANUFACTURING ENGINEERING LABORATORY AT MSFC

by

T. W. Lewis

Presented by

F. J. BEYERLE

Propulsion and Vehicle Engineering Laboratory
Marshall Space Flight Center
Huntsville, Alabama

The first clean room facilities at MSFC were developed for a tube cleaning operation. Shortly afterwards the clean room complex for a valve clinic was constructed. In addition to clean tubing, it was necessary to have a clean environment in which to assemble clean valves. The general layout of the clean room installation is shown in Figures 1-7.

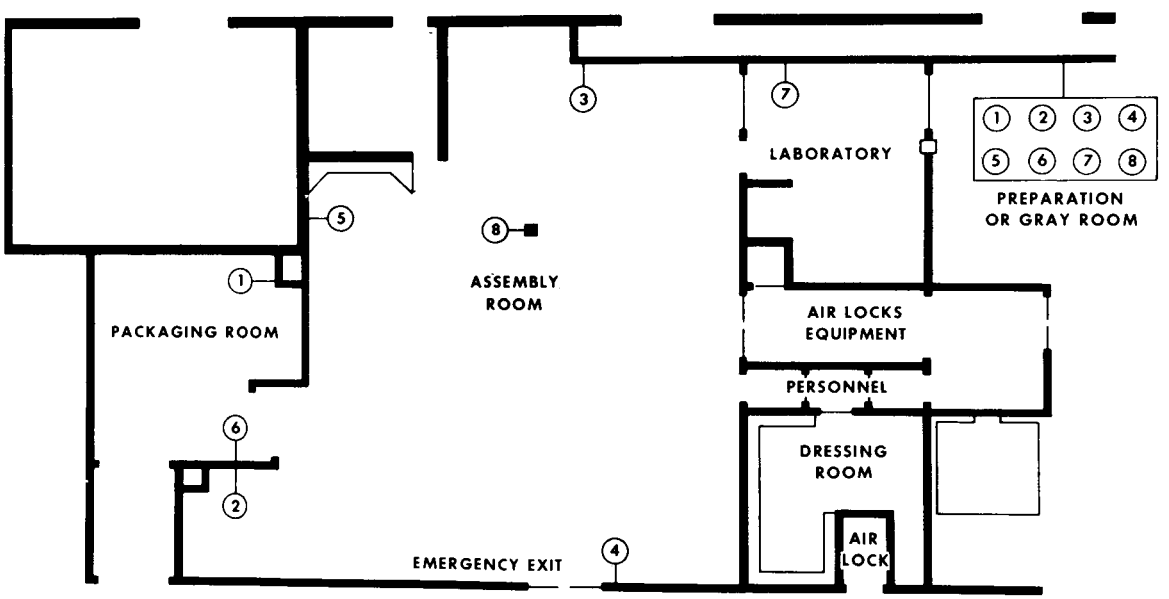



Figure 1. Valve clinic floor plan

Figure 2. Large valve (Note covered prints)

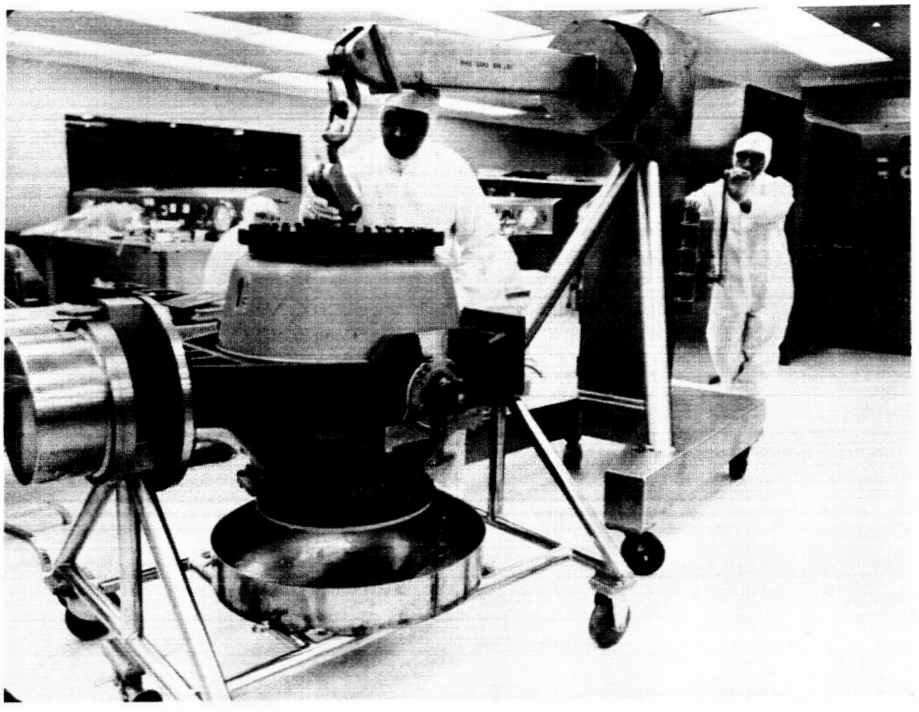


Figure 3. Large valve with crane

Figure 4. Small and intricate parts

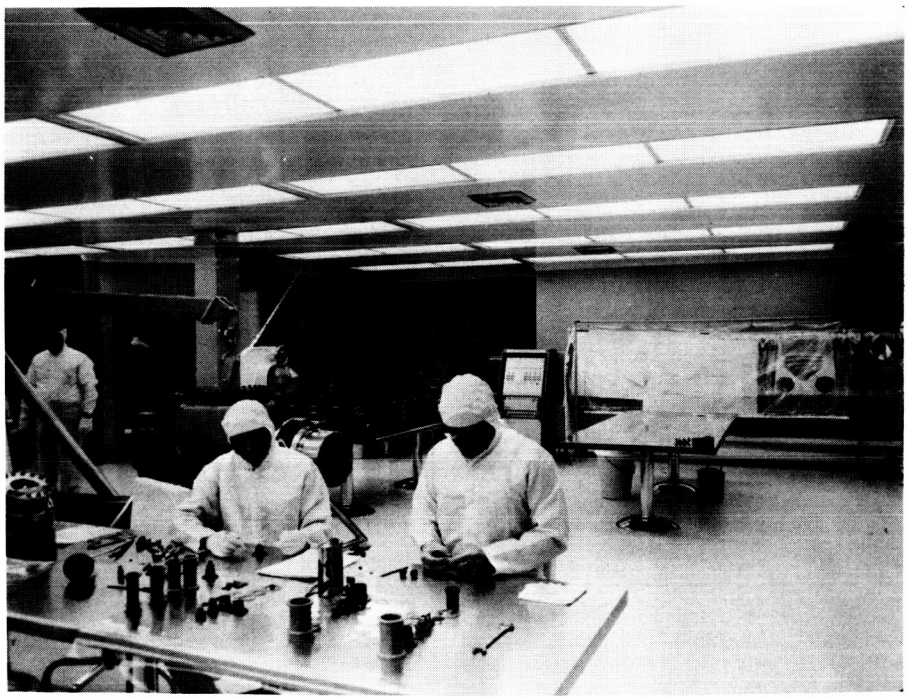


Figure 5. Overall view of lights, diffusers, and disposal cans

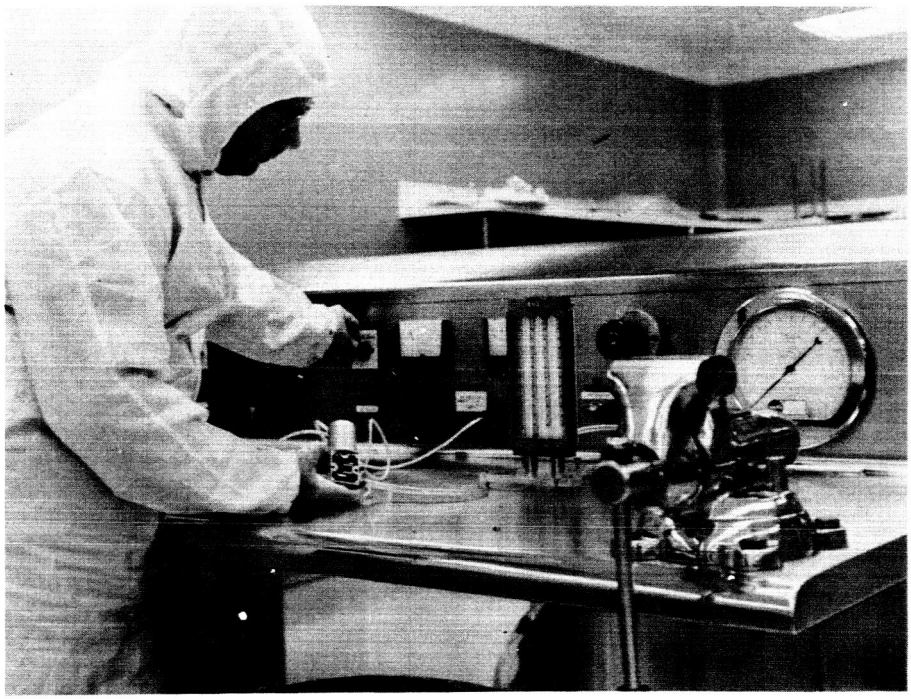


Figure 6. Checking apparatus

Figure 7. Writing bench

The basic principle in monitoring the environmental air is to draw a known volume of sample through the special membrane filter and to collect the airborne contamination on its surface. The particulate collected is then sized and counted under a microscope. The original sam ple should not have too many particles, but there must be a sufficient number collected to have them distributed uniformly over the whole filter in order to validate a statistical count.

The filter membrane was a German development during World War II. It was perfected for gas mask filters when the use of aerosols was developed for carrying chemical and biological warfare agents. These cellulose filters have unique characteristics, one of them being their similarity to a screen sieve because of their uniform pore size. This, in effect, gives them a property of being a "Go-No Go" counter. All particles below a specific size pass through the filter, and all those above that size are retained on the filter surface, where it may be inspected visually.

Oblique incident light is used to examine the filters because they are opaque and because shadows aid in the visual examination of the particles. The filters are gridded and are printed criss-cross with squares equal to one/one hundredth of the actual filtering surface. The microscope used gives 40 magnifications, which is a field within one grid square. The filter is placed so that the grids are square with the stage micrometer. At first, the whole filter is scanned by manipulating the stage micrometer to (1) count the fibers and large particles, (2) check the filter to make certain it is not torn or cracked, (3) make certain the fibers are uniformly distributed, and (4) ascertain whether it is practical to count particles statistically.

Monitoring was a patience-trying, time-consuming, and, sometimes, exasperating operation (see Figure 8). When the automatic particle counters appeared, it was considered a breakthrough (see Figure 9). However, under field conditions, it was almost impossible to get the microscopist's counts to agree with the data of the automatic instruments. As Phillip Austin's publications became better known, as Stoke's law was applied to the aerosol particulate, and as the data on many thousands of counts were accumulated, the particle size distribution curves were developed, accepted, and then published in Air Force Technical Order 00-25-203 and Federal Standard 209.

Many have pondered the reasons behind establishing the limits quoted by the specifications at the five micron ($5\mu$) and the five-tenths micron ($0.5\mu$) particle sizes. Since the five micron size is the smallest practical size that can be counted microscopically (magnification is the controlling factor), it was a good limiting point. The half-micron ($0.5\mu$) limitation was probably established so that the specification would not become obsolete immediately. The HEPA filters, when first developed, filtered out particles down to that size. Now they can filter down to the 0.3 micron size. An additional reason may have been that the straight line produced on log-log paper started to break in that vicinity. Also, there are too few particles in the five micron size range in a controlled environment for an automatic particle device to count properly.

The most popular automatic aerosol particle counter uses the 90° light scattering principle. The sample, drawn vertically through the sampling tube, passes through an intense horizontal light beam. A

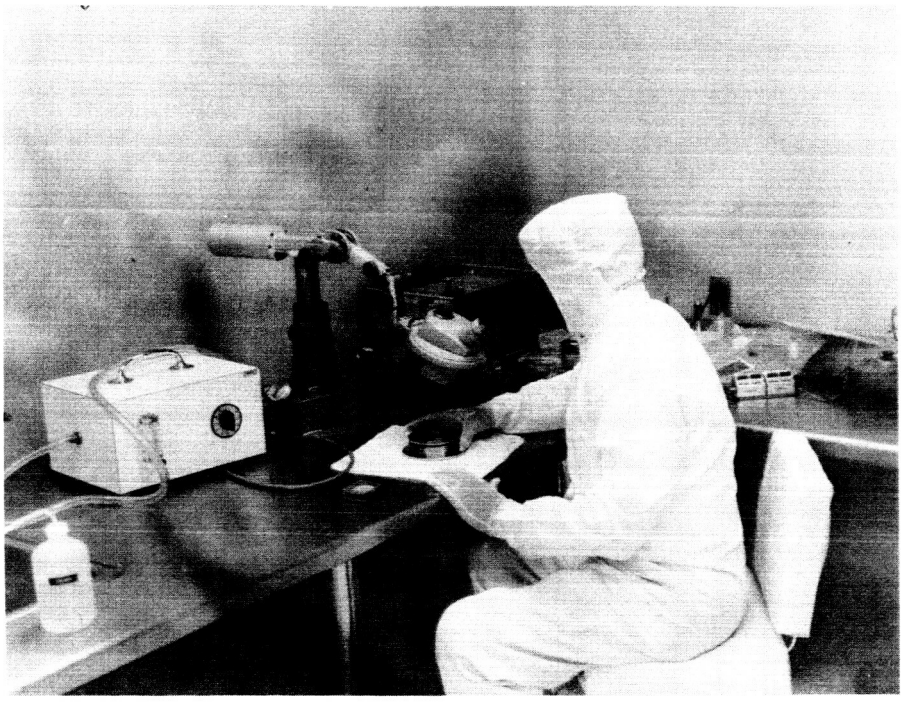


Figure 8. Monitor in clean laboratory

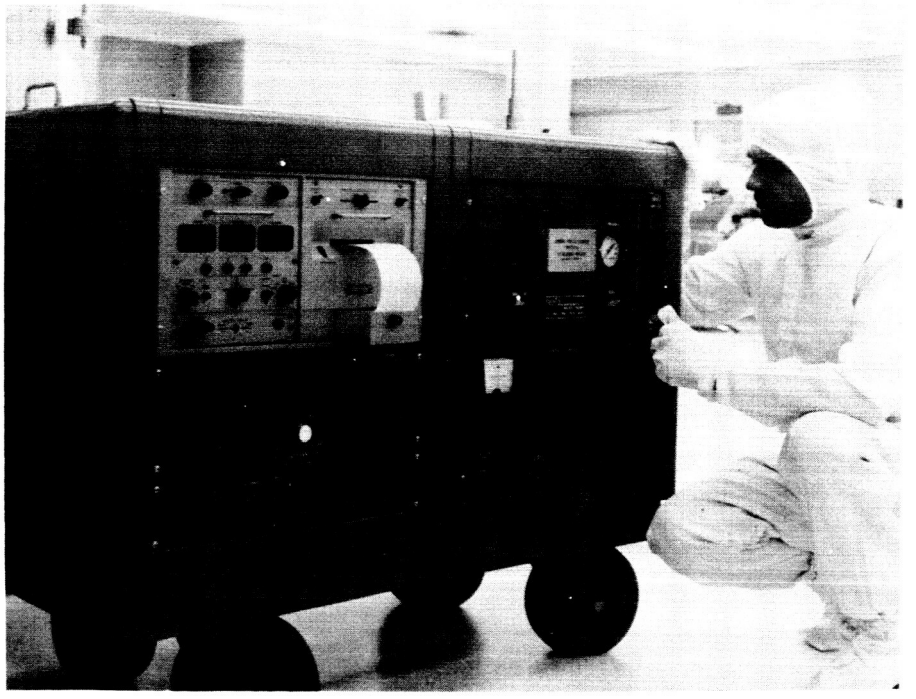


Figure 9. Standard particle counter

photomultiplier tube placed at 90° to both observes the light scattered toward it. The instrument samples approximately 200 ml per minute. Because of the disagreements with the microscopist's counts, it was wondered whether a representative sample was being counted. This posed a problem that can be expressed thusly:

$$\frac{\text{Particles in Sample}}{\text{Particle in Room}} = \frac{\text{Area of Sampling Tube}}{\text{Area of Room}} \times \frac{\text{Vol. of Sample}}{\text{Vol. of Room}} \times \frac{\text{Vol. of Sample Scanned}}{\text{Vol. of Sample}}$$

It might seem that a larger sample would overcome the difficulties.

A research contract for improvements was initiated with IIT, and it modified the automatic instrument as follows:

1. The flow rate was increased.
2. The air sheath principle was introduced.
3. The viewing volume was modified.

The observation of the light scattering principle might be likened to the seeing confetti tossed into the light beam of the projector. Assume that particles scatter light quantitatively in direct proportion to their size. The photomultiplier tube, located at 90° to the sampling tube and the light source, will receive the signals from the light scattered by the particles and then will size as well as count them. There is a certain amount of current oscillation greater than the carrier wave, and static is introduced from extraneous sources. This background static, commonly referred to as "noise," increases in direct proportion to the increase in the voltage. To count the smaller particles, it was necessary to increase the voltage. This made it increasingly difficult for the instrument to differentiate the signal from the background "noise," and the result was erroneously high particle counts. It was found that the instrument could not count particles smaller than 0.75 micron because of this signal/noise factor.

Difficulties in maintenance were encountered. The instrument was left on overnight when it was endeavored to get a count around the clock. The motors overheated and the plastic tubing melted and collapsed. The melted tubing finally stopped the flow of air and the pump motor burned out. The plastic tubing has been replaced by a stainless steel type, but this change made other adjustments necessary.

These studies demonstrated several valuable characteristics of the counter. It was a good indicator of the contamination present in the air. The data showed the following:

1. The count increased as the number of people present in the room increased.
2. The rooms had remarkable recovery characteristics.
3. The count was the highest when the janitors were cleaning.
4. Some of the overalls were wearing out and shedding excess lint.

The particle counter had proven useful, but still more improvement was needed. So another contract was given to IIT. Three separate sampling units were built for location in each of the three rooms in the

valve clinic (see Figure 10). All the data are now sent to a central data recording instrument located in a gray room (see Figures 11-13).

The instruments have been in operation sufficiently long to indicate that more improvements are required, especially the following:

1. A lower sampling height, preferably at table height.
2. A simpler lamp adjustment procedure.
3. A lower signal/noise ratio to allow counting in the 0.5-micron size range.
4. Printed circuit boards that do not require as much attention as the present ones.
5. Remove the sources of extraneous signals that reduce confidence in the data.
6. A simpler way of data presentation.
7. A fail-safe alarm that would indicate when an area is approaching excessive contamination.

MSFC still considers automatic particle counters of clean room air as being in a research and development status, one that requires improvements to give rapid, reliable, and reproducible results to meet the requirements imposed by the specifications.

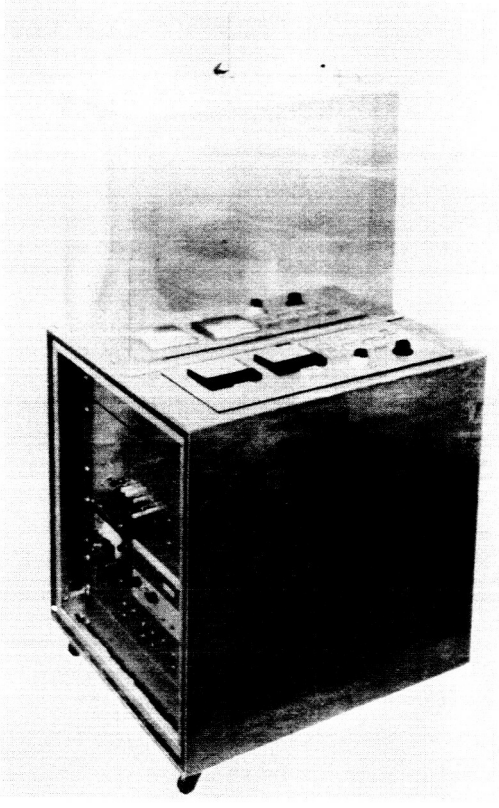


Figure 10.
Particle counter air sampler

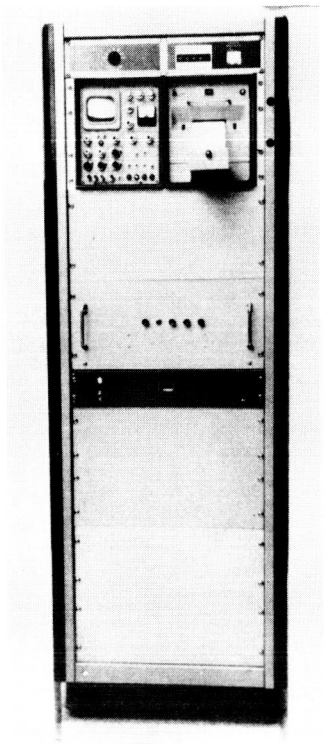


Figure 11.
Data center

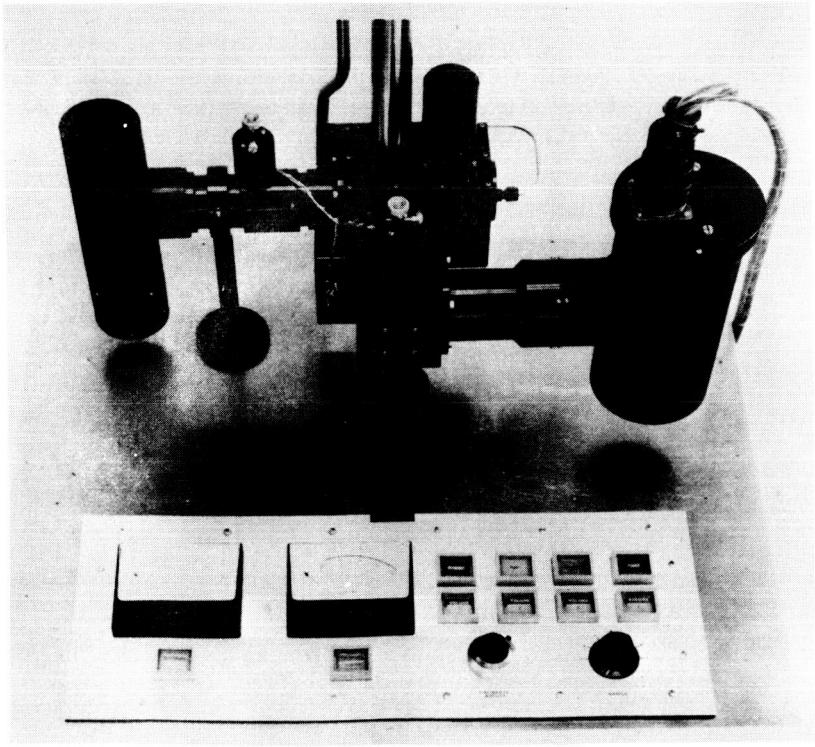


Figure 12.
Sampler optics

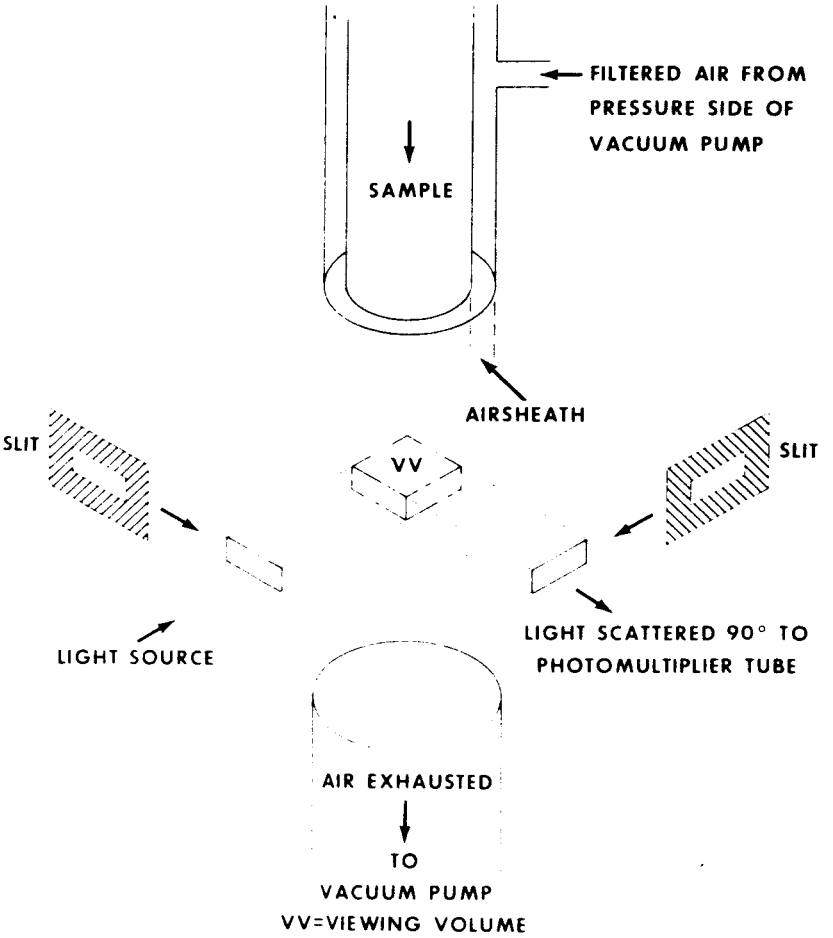



Figure 13. Particle counter optics with 90° light scatter

# 2. AUTOMATIC AND REMOTE MONITORING SYSTEMS FOR AIRBORNE PARTICLES

by

A. LIEBERMAN

IIT Research Institute
Chicago, Illinois

The problems of monitoring airborne particles involve a number of steps. First, a sample must be taken from the atmosphere where the particles are present. The sample must be transported to an analytical device where the particles in the air are analyzed for size distribution and concentration. Next, the data from the analysis are reduced and interpreted. Finally, a decision is made regarding the concentration and size distribution function of the airborne particles.

A number of problems exist in carrying out these steps. First, are representative samples obtained from the atmosphere? Figure 1 shows errors which can arise from anisokinetic sampling. Next, how well does the sampling handling system operate, that is, are materials lost or distorted in transit between the ambient environment and the analytical system. Figure 2 shows duct losses in turbulent flow. Next, how well does the analytical system operate; is it stable, is it accurate, does it record the correct size parameter for the problem of concern? Next, how acceptable are the data; do they have meaning in discussing the size distribution, are there sufficient data for statistical reliability, have coincidence effects been taken into consideration? Finally, how are the data stored, recorded, or acted upon?

A number of methods permit sampling, analyzing, and recording particles in airborne systems. Methods of recording particle measurements are based on specific properties of the particles, such as electrical charge, mass, light scattering, or radiation absorption. Therefore, rather than detail instruments and measurement methods which are not applicable to the problem of monitoring contamination for modern day assembly technology, this discussion will be restricted to the method used for conventional analysis of clean rooms and contamination levels in sensitive areas.

The primary instrumental method of observing particulate contamination in these areas is that employing light scattering. In light scattering devices, particles are drawn into an illuminated viewing area. As they pass through, each particle produces a single pulse whose light flux is a direct function of the projected area of the particles. Thus, pulses are produced at a fairly rapid rate. They are capable of electrical summing, recording, and the usual data processing associated with pulse generating systems.

General design criteria are essentially the same for most single particle counting systems. Three divisions of operations are concerned.

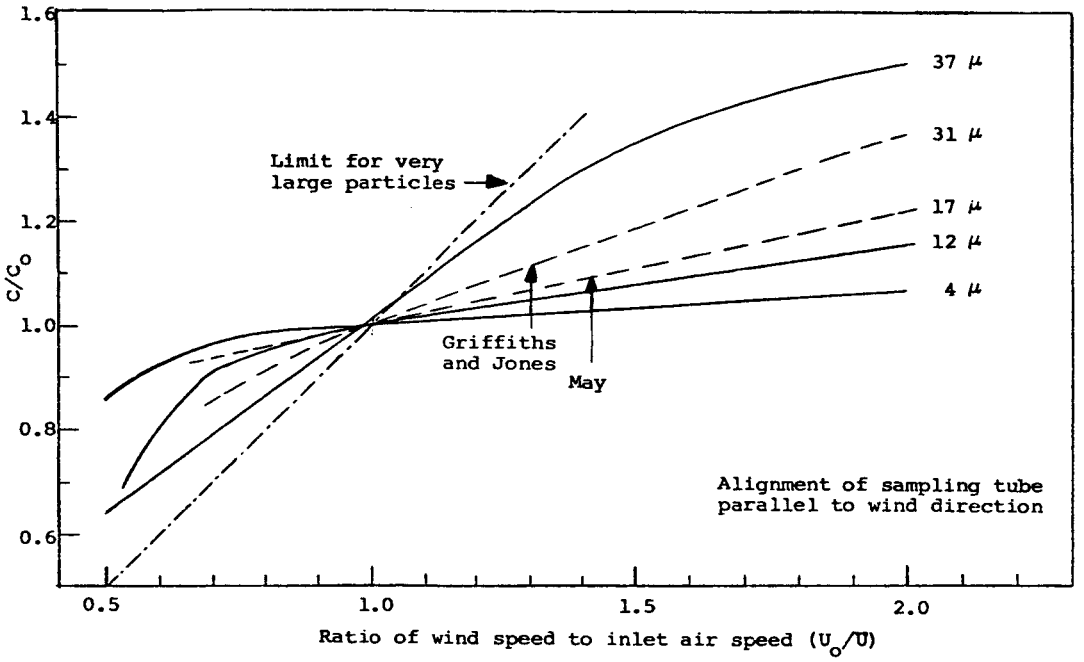



Figure 1. Anisokinetic sampling error

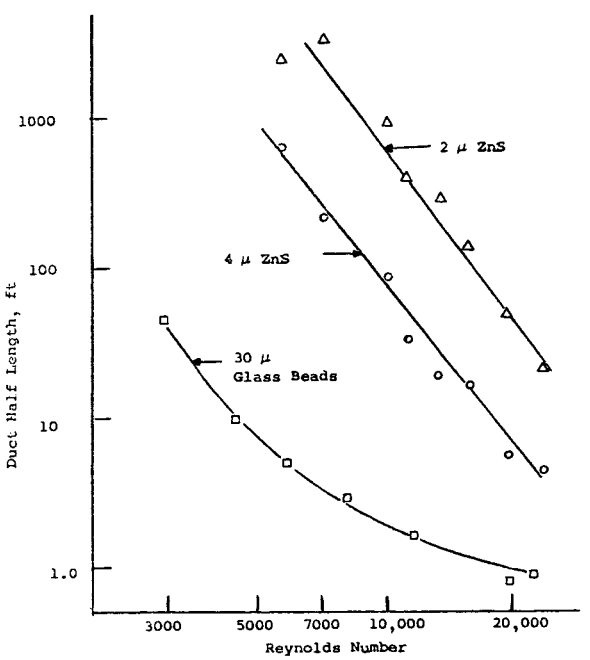



Figure 2. Duct half-length for particle deposition in 1.0-in. aluminum pipe

The first is an aerosol control system which samples and transports a representative portion of the sample to a sensing zone. Figure 3 shows an aerosol control system used to reduce wall losses and vaporizing effects. The second is an optical section which provides a light source to illuminate the particles and a detector to convert the light impulses to electrical impulses. Figure 4 shows an optical system for right angle scattering measurements. The third is an electrical control system to classify the generated electrical impulses in terms of particle size and number and permit transformation of the raw data into usable information. Figure 5 shows a simplified block diagram of a working pulse height analyzer.

The aerosol control section performs the following functions. It samples the aerosol, transports the particles to the sensing zone, eliminates losses, and avoids size discrimination in handling the particles. The optical section illuminates a portion of the aerosol stream and collects the light scattered by the particles in this region. The optical section must produce a uniformly illuminated and defined volume of space in which no more than one particle is present an acceptably small portion of the total time. The optical system must transmit the scattered light pulses to the detector with good fidelity. The optical system should also contain self-calibration capabilities; that is, a capability for simulation of aerosols with a known signal. The electrical system must contain controls for operation and activation of the optics and air handling mechanical systems. It must reproduce the light pulses faithfully as electrical signals. The electrical system must provide data that are capable of being handled, transmitted, and controllable. Figure 6 shows a unit used at IIT for repetition measurements to study cloud decay.

IIT has recently developed two systems which use light scattering and are capable of automatic and remote operation. The first was developed on contract for Marshall Space Flight Center. The system, at this time, consists of three light scattering air handling systems whose outputs are multiplexed into a single data center. The operating specifications of this system are as follows. Particulate matter is to be monitored in air simultaneously in three separate locations. The analysis of particulate concentrations and size distributions is to be available outside the clean room for immediate use. Particles in discrete ranges from 0.5$\mu$ to greater than 100$\mu$ have to be monitored. In addition, high volumetric sample flow-rate capability is desired. Variable sampling periods are to be built in. Simultaneous counting and categorizing into sizes of all particles are required. A minimum number of electrical noise spurious counts is permitted. The capability of reaching a statistically significant number of counts in all size ranges is necessary and maximum mobility and liability are desired.

Figure 7 is a drawing which shows the transducer head; Figure 8, a photograph of the head in place on the air handling optical unit; and Figure 9, the single pump flow control system that is used. The concentric tube provides an air sheath for the aerosol particles to prevent the loss of particles into the clean sample chamber and to eliminate recirculation of particulate material. The air sample rate was 0.75 cfm with an air sheath flow rate of 2.25 cfm. Table 1 shows the characteristics of the air handling system.

By using a variable viewing volume controlled viewing zone principle, it is possible to vary the actual gas volume analyzed from .0076 cu ft/min to 0.42 cu ft/min. Table 2 shows the counting rate which would be seen in a Class 100,000 or a Class 10,000 clean room. In a Class 100

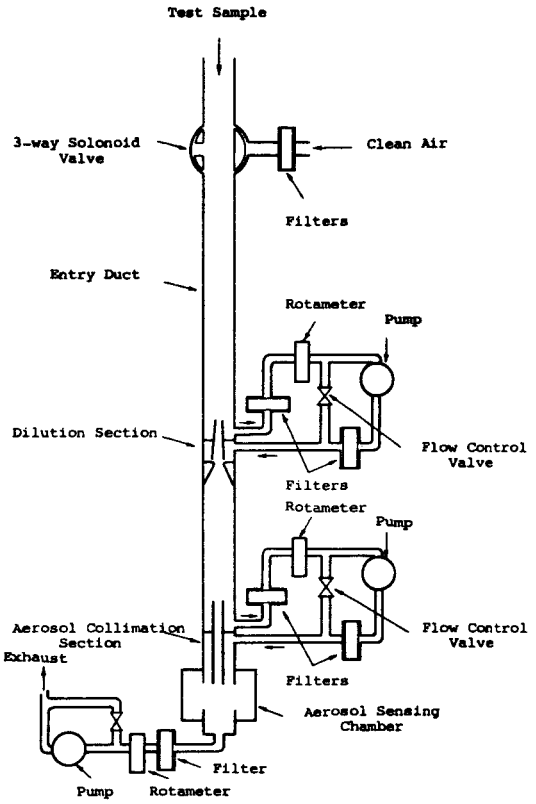
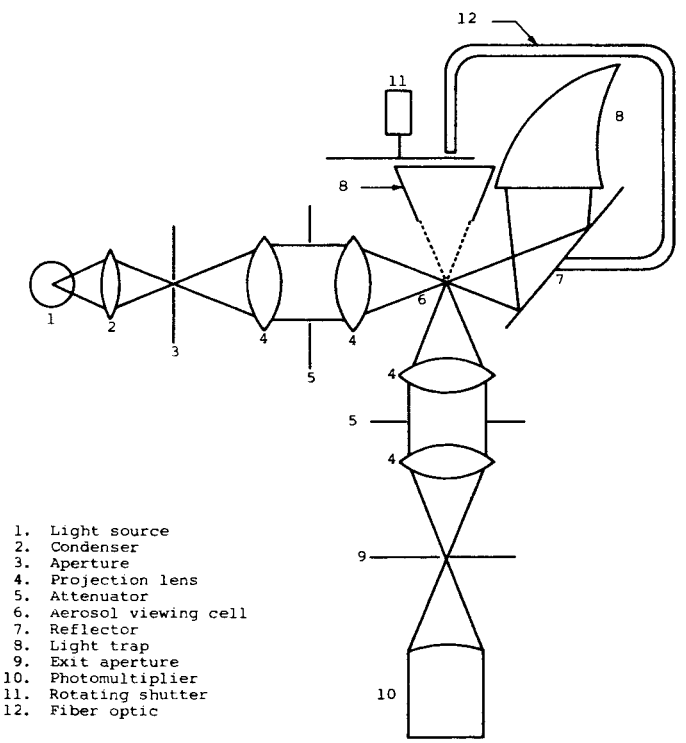



Figure 3.
Aerosol control section

Figure 4.
Schematic of optical system




1. Light source
2. Condenser
3. Aperture
4. Projection lens
5. Attenuator
6. Aerosol viewing cell
7. Reflector
8. Light trap
9. Exit aperture
10. Photomultiplier
11. Rotating shutter
12. Fiber optic

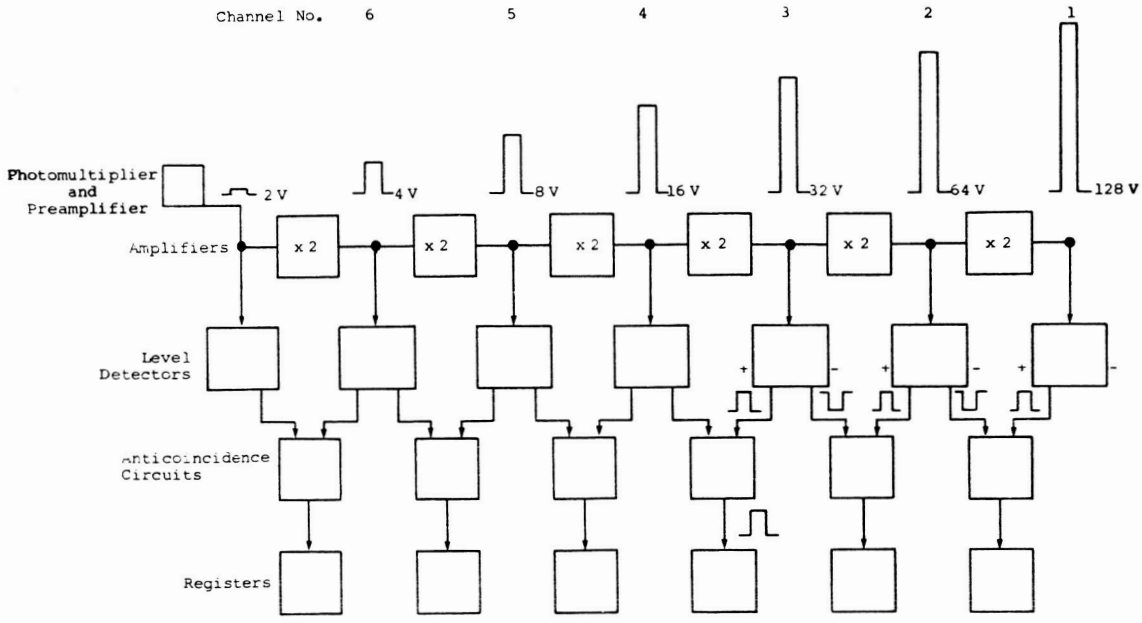



Figure 5. Electrical system - simplified block diagram

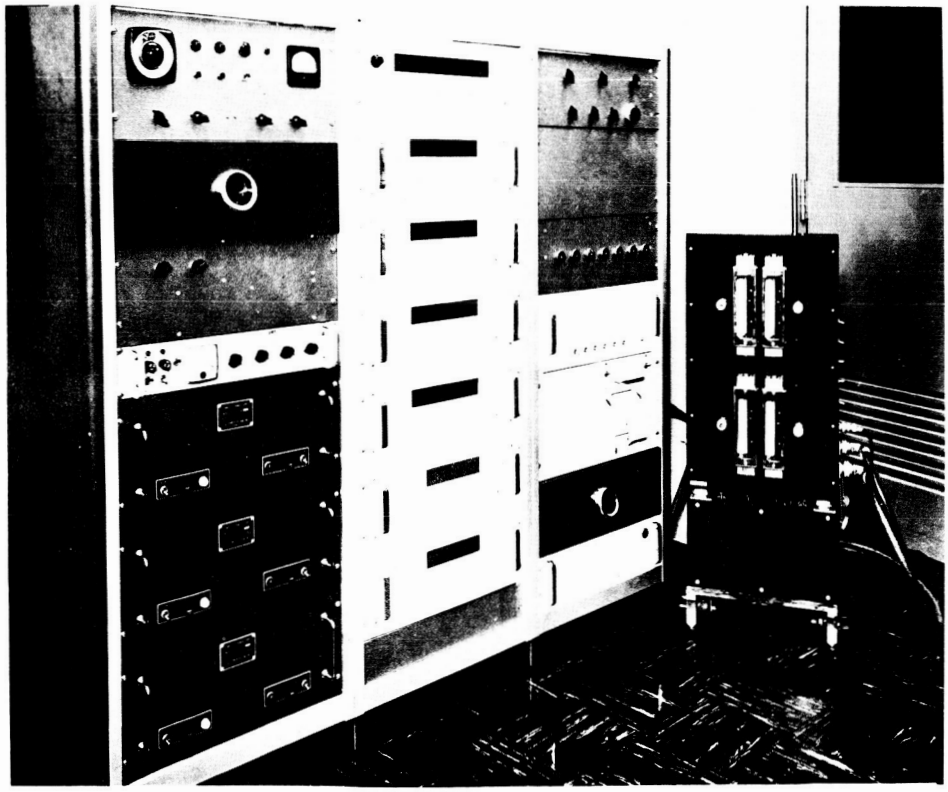


Figure 6. Light scattering particle counter

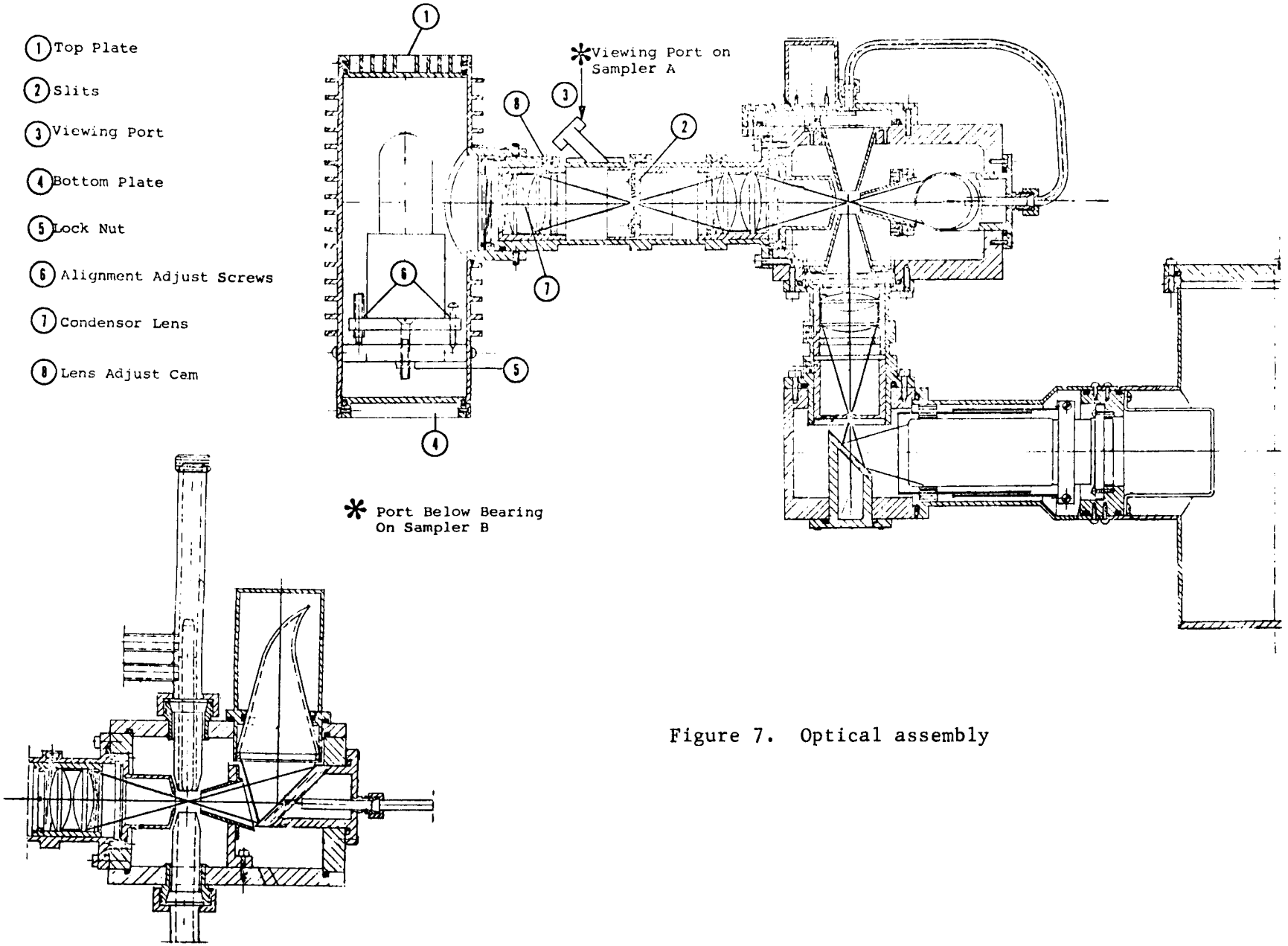



Figure 7. Optical assembly

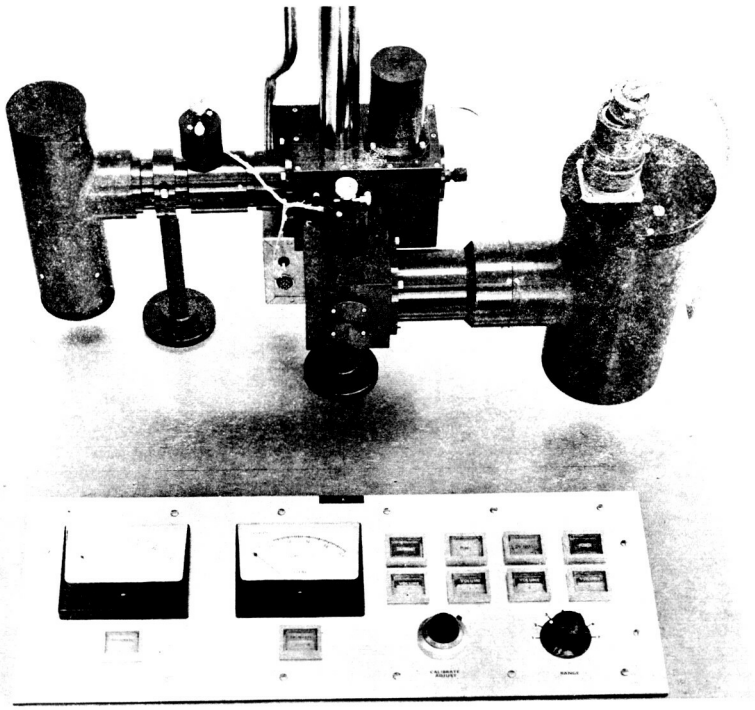


Figure 8. Head in place on air handling optical unit

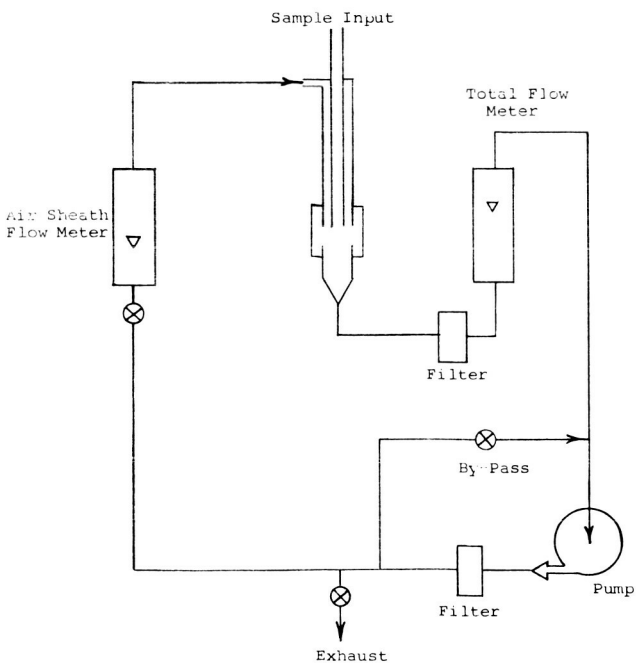



Figure 9. Air handling schematic

Table 1

AIR HANDLING PARAMETERS

| Sampling Tube | English Units | Metric Units |
|---|---|---|
| ID | 0.625 in. | 15.8 mm |
| Area | 0.307 $in.^2$ | 198. $mm^2$ |
| Flow rate | 0.75 cfm | 0.021 $m^3$/min. |
| Avg. linear vel. of air | 5.9 fps | 1.8 m/sec |
| Max. linear vel. of air | 11.8 fps | 3.6 m/sec |
| **Air Sheath Tube** | | |
| ID | 1.25 in. | 31.8 mm |
| Area (less sample tube area) | 0.913 $in.^2$ | 590. $mm^2$ |
| Flow rate | 2.25 cfm | 0.064 $m^3$/min. |
| Avg. linear vel. | 5.9 fps | 1.8 m/sec |

**Sample Analyzed**

| Viewing Zone, mm | cfm | % Analyzed |
|---|---|---|
| 1 x 1 x 1 | 0.0076 | 1.0 |
| 2 x 2 x 1 | 0.0302 | 4.0 |
| 4 x 4 x 1 | 0.1175 | 15.7 |
| 8 x 8 x 1 | 0.424 | 56.6 |

**Count Rates**

Class 100,000 Clean Room

100,000 particles/$ft^3$ > 0.5 $\mu$
700 particles/$ft^3$ > 5.0 $\mu$

Class 10,000 Clean Room

10,000 particles/$ft^3$ < 0.5 $\mu$
65 particles/$ft^3$ > 5.0 $\mu$

Table 2

COUNTING RATES IN CLASS 100,000 AND 10,000 CLEAN ROOM

**Class 100,000**

| Viewing Zone, mm | Counts/min | |
|---|---|---|
| | 0.5 $\mu$ | 5.0 $\mu$ |
| 1 x 1 x 1 | 765 | 5 |
| 2 x 2 x 1 | | 21 |
| 4 x 4 x 1 | | 82 |
| 8 x 8 x 1 | | 296 |

**Class 10,000**

| Viewing Zone, mm | Counts/min | |
|---|---|---|
| | 0.5 $\mu$ | 5.0 $\mu$ |
| 1 x 1 x 1 | 71 | 0.46 |
| 2 x 2 x 1 | | 2 |
| 4 x 4 x 1 | | 8 |
| 8 x 8 x 1 | | 27 |

**Pulse Times**

| Viewing Zone, mm | Time, $\mu$/sec | | |
|---|---|---|---|
| | Max. | Min. | Avg. |
| 1 x 1 x 1 | No significant variation | | 280 |
| 2 x 2 x 1 | 285 | 280 | 282 |
| 4 x 4 x 1 | 296 | 280 | 289 |
| 8 x 8 x 1 | 375 | 280 | 320 |

clean room, approximately 40 counts/min would be received for particles half micron and larger when the 8 x 8 x 1 mm slit size in used. Thus, an approach to statistically significant count rates can be achieved. Figure 10 shows coincidence errors that can be expected for several viewing volumes, assuming concentrations as shown.

The electronic system consists of a pulse height analyzer (Figure 11) which measures the size of the pulses produced as each particle passes through the viewing zone. The photomultiplier output current enters the summing point of a current-to-voltage converter, which consists of an operational amplifier, first stage. This input stage is followed by three identical operational amplifier stages, each with a voltage gain of -16. The output of the last voltage amplifier contains a nonlinear feedback network which supplies current to the input of the first stage. This feedback permits extremely high stability and low drift. The outputs of the pulse amplifiers are directed to threshold analyzers which sample and measure the pulse amplitudes. This configuration permits the use of several comparators operating at different threshold voltage levels from the same reference, each of which produces an output pulse for one and only one particle size.

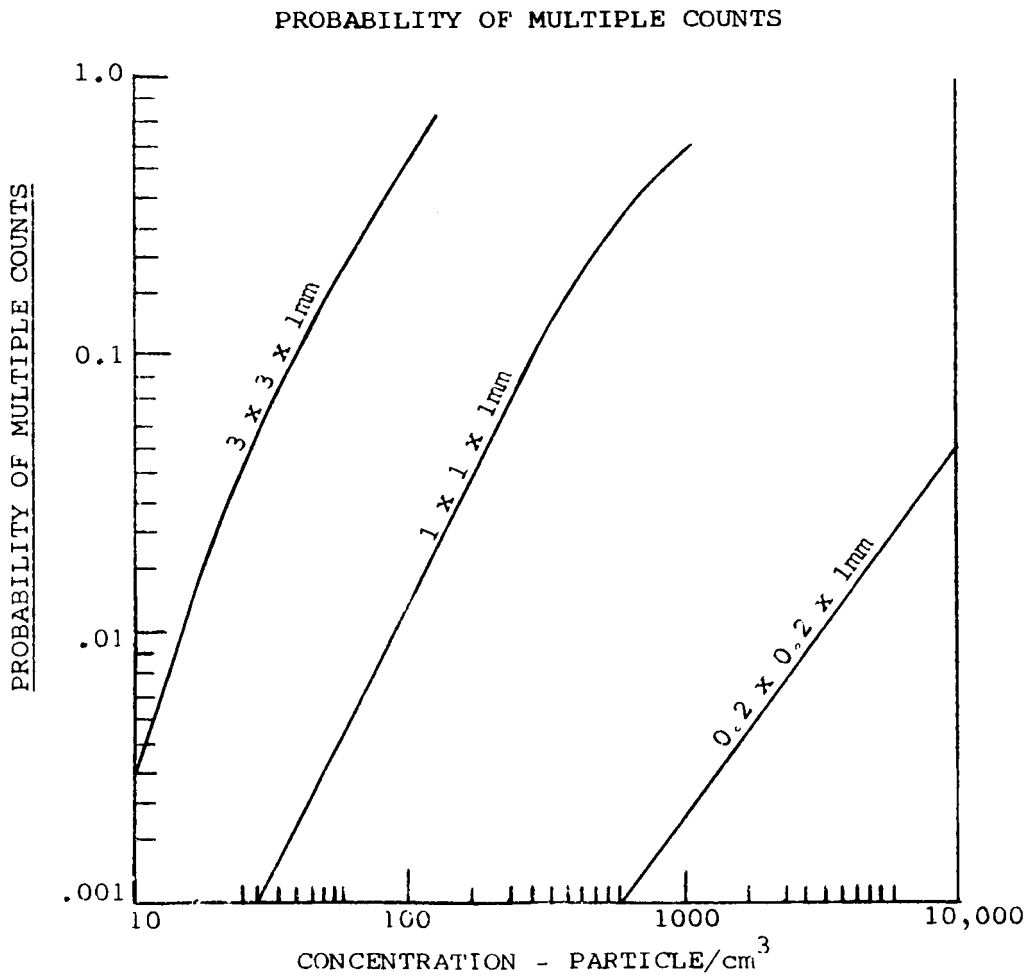



Figure 10. Coincidence errors for several viewing volumes

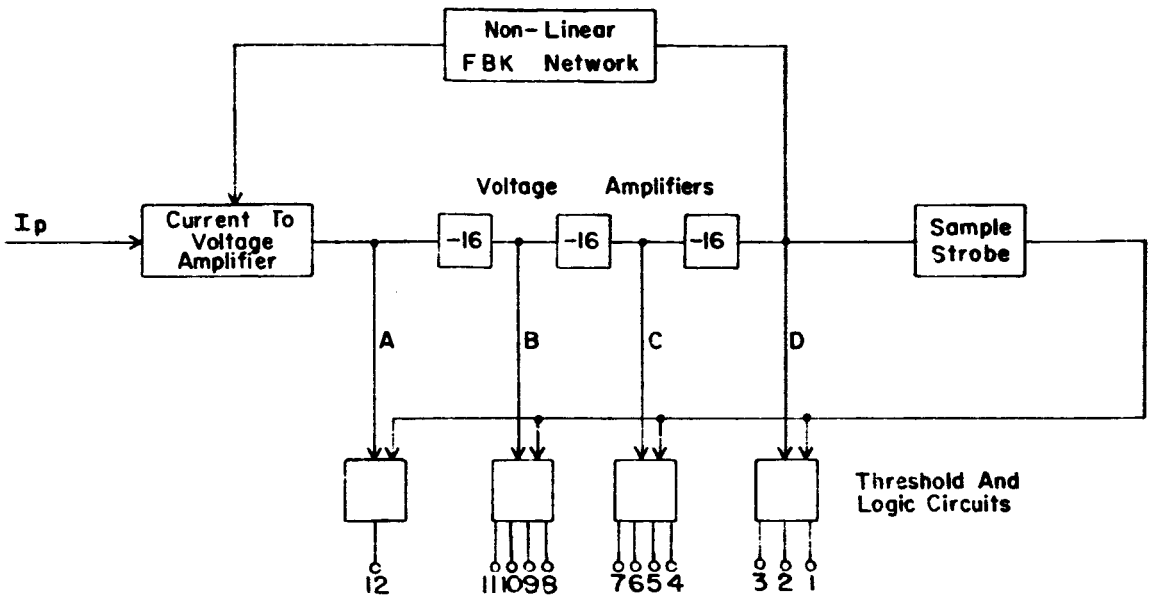



Figure 11. Pulse height analyzer

The data system shown in Figure 12 accepts particle count data from the three remote samplers, transmits the count data by wire to the data center, enters it into a memory unit, and makes periodic printout of the accumulated particle totals. The data originate in the pulse height analyzer at each sampler in the form of pulses of standard amplitude on output lines corresponding to the 11 available size categories. A manually operated switch selects six adjacent lines for transfer to a diode encoder which translates the input pulse on one of the six lines to a combination of simultaneous pulses on three output lines according to a straight binary code. These pulses are sent to the data center. The pulses at the data center are then entered into a memory unit by a digital logic unit. Associated with the logic unit is a clock programmer which controls the automatic printout of accumulated data. At preselected intervals, accumulation of data is temporarily halted and the contents of the memory unit are read out and printed on paper tape together with time of day. Once the printout is completed, the data center returns to data accumulation process.

The memory unit has 100 channels of core storage, each channel capable of storing any number from 0 to 99,999. The memory unit operates like a stepping switch; the count in a particular storage channel is increased each time a count of given size arrives at the data center from the sampler, each count representing 1 particle. The memory has 18 channels assigned to three samplers, (six to each sampler), and each sampler tallies the number of events occurring in each category (the particle size range).

The clock programmer continually monitors the channel number of the data that are being sent from the memory to the printer. When a preset time interval has been attained, a pulse is transmitted that takes effect only when the system is not scanning. If the system is in the middle of a scan when the pulse is received, the scan is completed before the time input pulse takes effect. This input pulse inhibits the entry of further data into the system, stops further scans, and initiates a readout command to the memory unit and its associated printer. This command

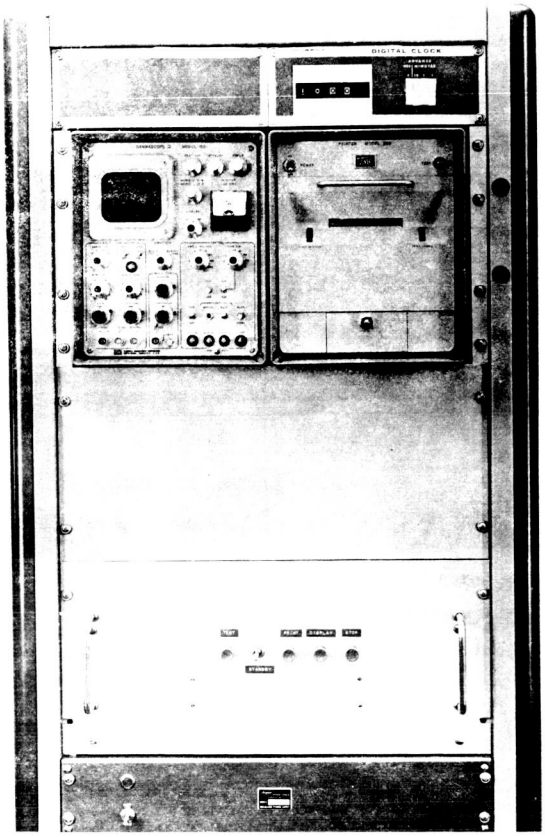


Figure 12.
Data center

causes the memory to step through its channels and presents to the print-out unit the data that is stored.

The unit was calibrated with four separate materials. These are (1) air cleaner test dust in a size range of 1/2 to 15$\mu$, (2) resin microballoons in a size range of 5 to 100$\mu$, (3) saccharin in a size range of 0.5 to 15$\mu$, and (4) glass beads in a size range of 5 to 80$\mu$. Figures 13 and 14 show typical calibration data for the samplers in which the recorded data is compared with microscopic measurement of particle size distribution. After the particle counters were delivered to NASA, they were operated in the atmosphere at the valve clinic. Table 3 shows the data obtained. The particle counter samples were obtained, after the sampler was calibrated for large particles, by counting in the size range of 4 to 128$\mu$ while membrane filter samplers at the same time were monitoring the particles in the air. The membrane filter samples indicate fluctuations in total number of particles above 5$\mu$ whereas the particle counters indicated only mild variations in the count. Variations in the data and a limited number of tests prevent valid conclusions. However, the two methods do show a tendency toward agreement in particle concentrations.

During the time that this work was being completed, an additional remote and automatic monitoring system was being developed on contract for the Army Dugway Proving Grounds. The objective of this program was development of a particle counter to sample isokinetically and remotely for particles in the size range of 1/2$\mu$ to 500$\mu$ plus. This sampling system must be capable of remote and automatically controlled operation and must produce data that can be used to show real time particle size distribution and concentration at 1 or more points in a test grid.

Table 3

PARTICLE CONCENTRATION IN AIR
OUTSIDE THE NASA VALVE CLINIC

| Test Series | Sampler | Time of Day | Sampling Period, min. | Particle Count/ft$^3$ Particle Counter[a] | Particle Count/ft$^3$ Membrane Filter[b] |
|---|---|---|---|---|---|
| 1 | A | 9:40 | 10 | 460 | |
| | | 9:45 | 2 | | 867 |
| | | 9:50 | 10 | 530 | 42 |
| | | 11:20 | 8 | 300 | 169 |
| | | 11:30 | 7 | 202 | |
| | | 11:40 | 20 | | 222 |
| | | 14:00 | 10 | 380 | 60 |
| | | 14:40 | 10 | 290 | 38 |
| 2 | A | | 30 | 480 | 350 |
| | | | | 330 | 660 |
| | B | | 30 | 760 | 350 |
| | | | | 640 | 660 |
| | C | | 30 | 350 | |
| | | | | 660 | |

[a]Size range of particle counter was 4 to 32 μ for Test Series 1, and 4 to 128 μ for Test Series 2.

[b]Size range of membrane filter was 5 to>100 μ.

A transducer system employing single particle counting by light scattering was used. Two sensors, one for particles 1/2$\mu$ to 32$\mu$, and one for particles 16$\mu$ to 500$\mu$ plus, are used to detect aerosols. Automatically controlled isokinetic sampling is used for Sensor A, e.g., 0.5$\mu$ to 32$\mu$ particles. Wind speed and wind direction sensors are used to control inlet pumping speed and inlet orientation for the sampling system. The sampling system for Sensor A samples horizontally and with variable inlet speed. The system is shown in Figure 15. Mass flowmeters

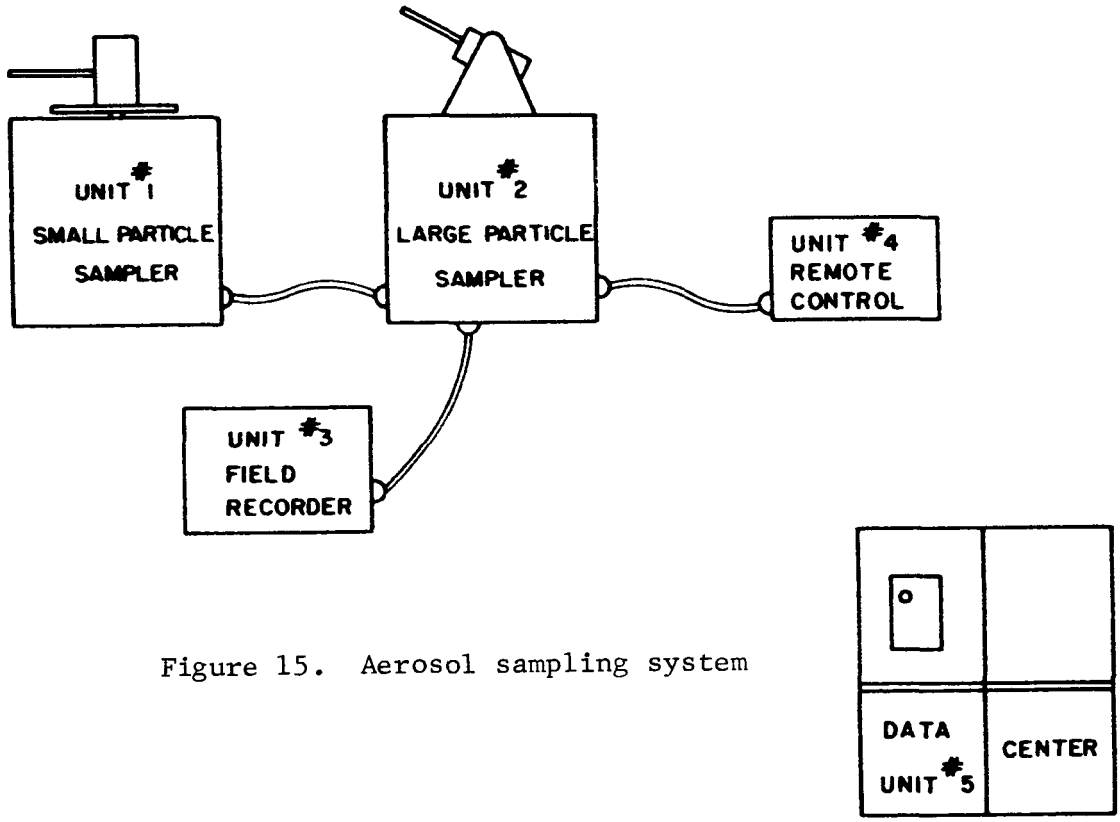



Figure 15. Aerosol sampling system

are used to insure constant exhaust for the system and variable input in accordance with wind speed measured by a rotating cup anemometer, as shown in Figure 16. The wind direction sensor is used to position a servo motor which orients the intake into the wind at all times. Signals from the wind speed sensor are stored and are used to indicate inlet sample quantity. Particle sizes are recorded in 8 channels where they are transferred to digital data for subsequent transmission to the data center.

The second field transducing system Sensor B is used to observe particles of 16$\mu$ to 500$\mu$ plus. This system samples at a constant inlet gas velocity and does not change orientation with wind velocity, and the particles to be sampled are sufficiently large to be considered capable of semiballistic trajectory. The pulse height analyzer in this unit, as in the A unit, transfers the data in terms of particle size distribution to eight channels of binary coded digital data for transmission to the data center. Sensor B is shown in Figure 17.

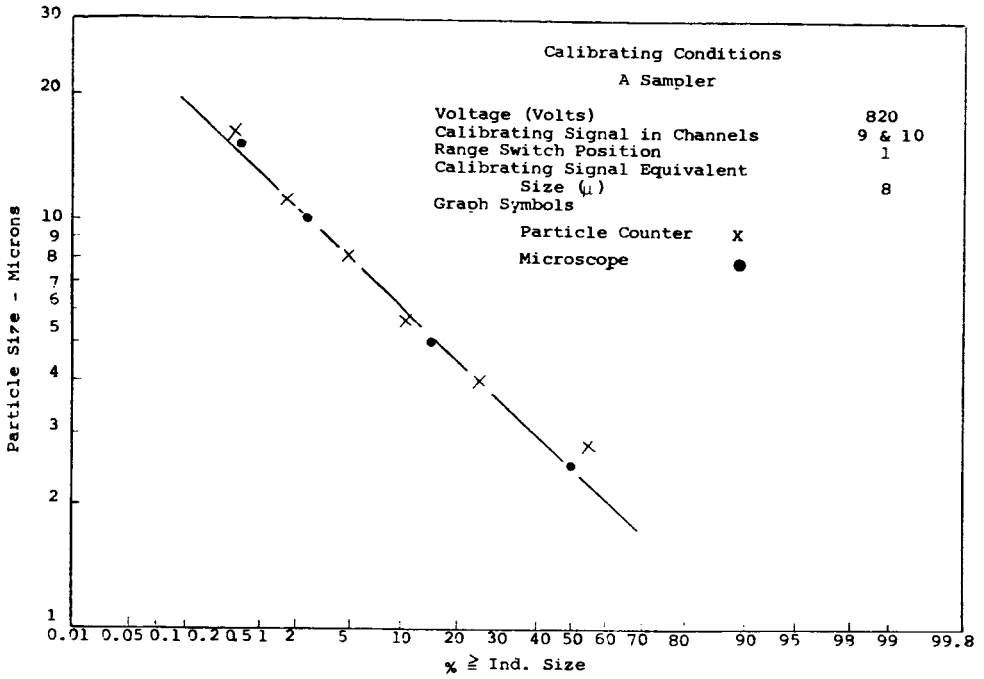



Figure 13. Particle counter calibration with ac test dust

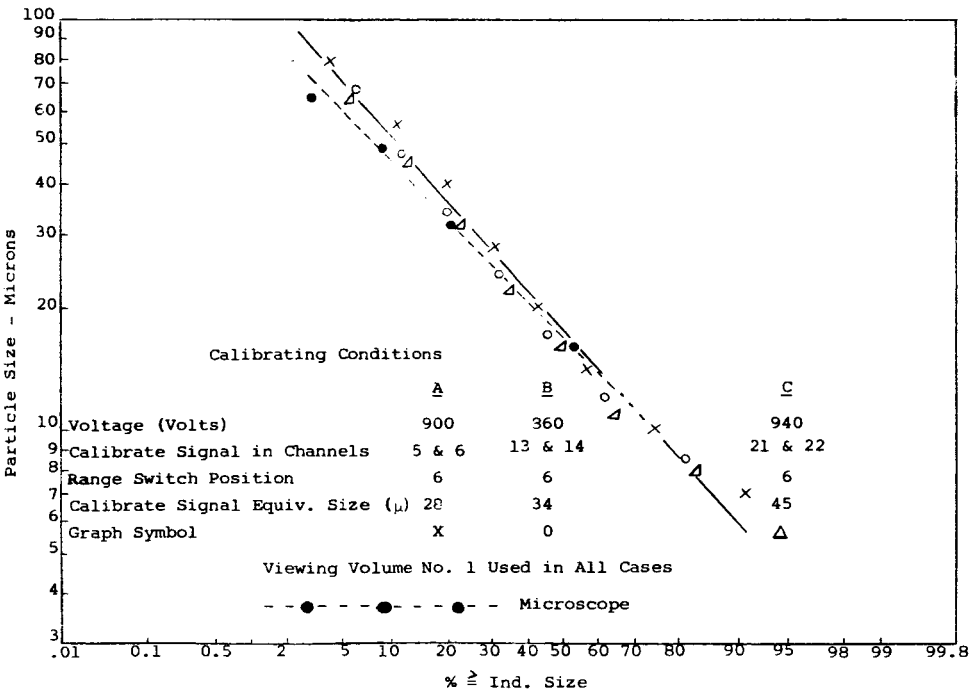



Figure 14. Particle counter calibration with microballoons

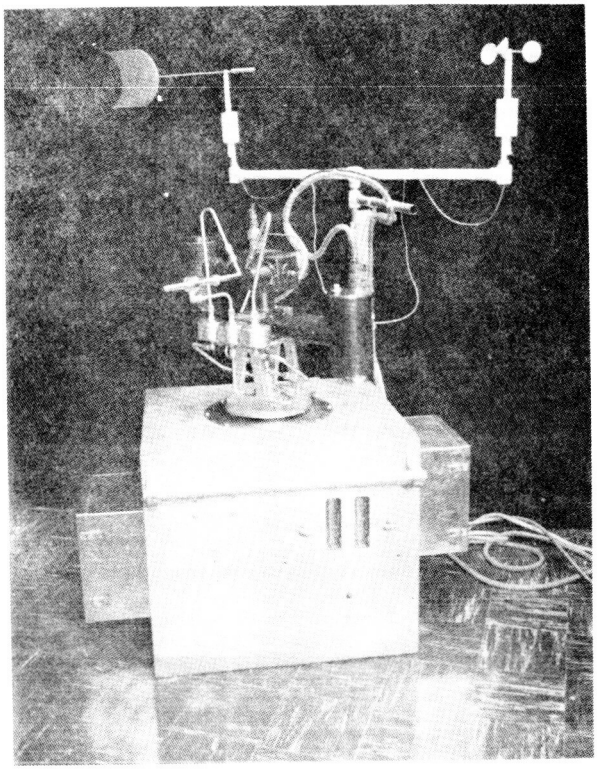


Figure 16.
Sampler A, Unit 1

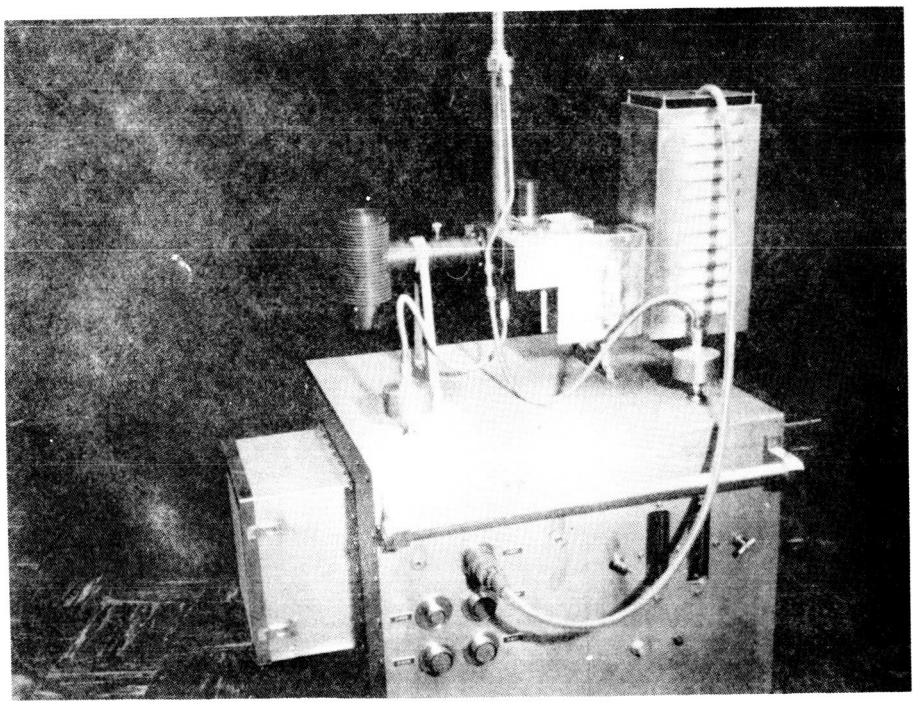


Figure 17. Sampler B, Unit 2

Since these two units are to be exposed to aerosols in the field under conditions which make it impossible for personnel to make adjustments during the course of a test, it is necessary to use a remote control unit to operate the field sensors (Figure 18). This unit performs two functions. The first is to send, one at a time, any of a number of discrete messages as commands to start or vary any of the functions of the samplers, such as pump speed, photomultiplier dynode voltage, and calibration position, and to indicate the status of several devices at the sampler.

The remote control unit is adaptable to either radio or wire transmission links. Since rapid response to commands is not necessary and maximum reliability and simplicity are necessary, a tone control system is used. Groups of tone generators are all connected into one pair of telephone lines by individual switches. At the sampler stations, resonant reed switches are arranged in parallel across the line and are tuned to frequencies that correspond to those at the remote control unit. The resonant reed switch, when closed by a tone generation, actuates a relay. By sending more than one tone, it is possible to increase the number of messages by considering that the presence or absence of tones corresponds to ones and zeros of a binary number. At this time the tone generation system transmits the following commands to the remote sensors:

1. Off, on
2. Start recording
3. Standby
4. Mark Beginning of test
5. Operate Sensor A
6. Operate Sensor B
7. Set Sensor A flow control to automatic
8. Set A or B Sensor flow at any of four fixed rates
9. Show wind speed
10. Show wind azimuth
11. Calibrate (the wind speed and wind azimuth meters are interchangeably connected to the calibrate pulse rate indicators. For calibration it is necessary that the pulses from two adjacent channels be at the same rate; thus the two meters must show approximately the same reading).
12. Lamp on
13. Lamp level control
14. Sensitivity increase for both A and B sensors
15. Sensitivity decrease for both A and B sensors

Controls 13 through 15 are fixed range potentiometer motor drive units.

For operation, it has been found necessary to show integrity of the transmission line. Thus, a responding circuit is plugged in so that each command signal transmitted produces a continuity check which shows as "Command Received" on the remote control unit.

The next field unit is the field electronics station (Figure 19). This station receives data on punched paper tape from remote Sensors A and B and from the meteorological network. The data received and

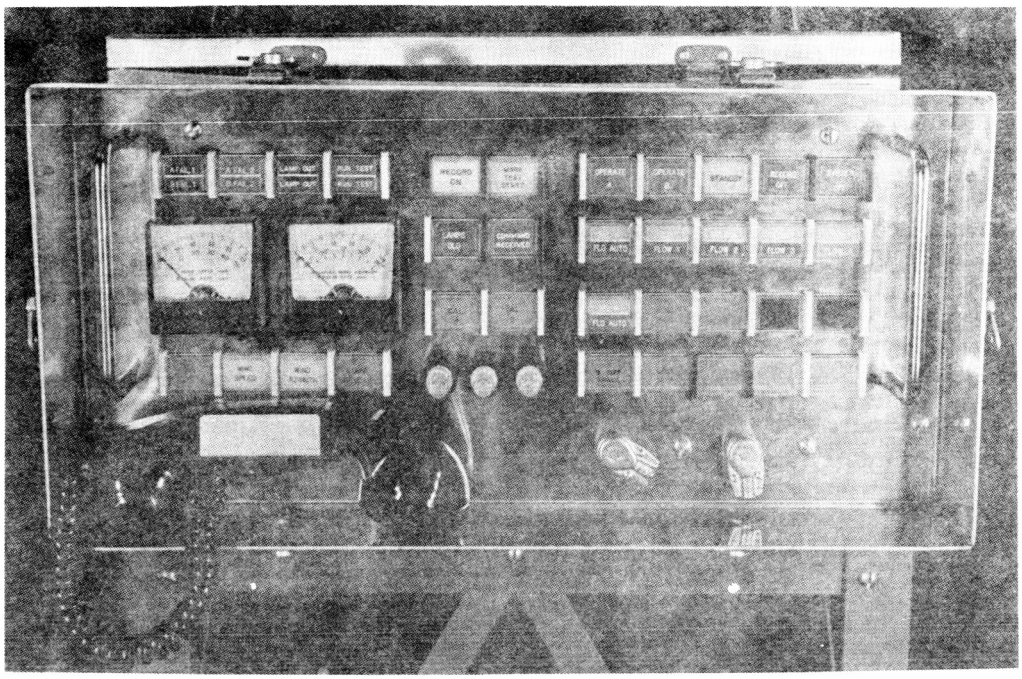


Figure 18. Remote control Unit 4

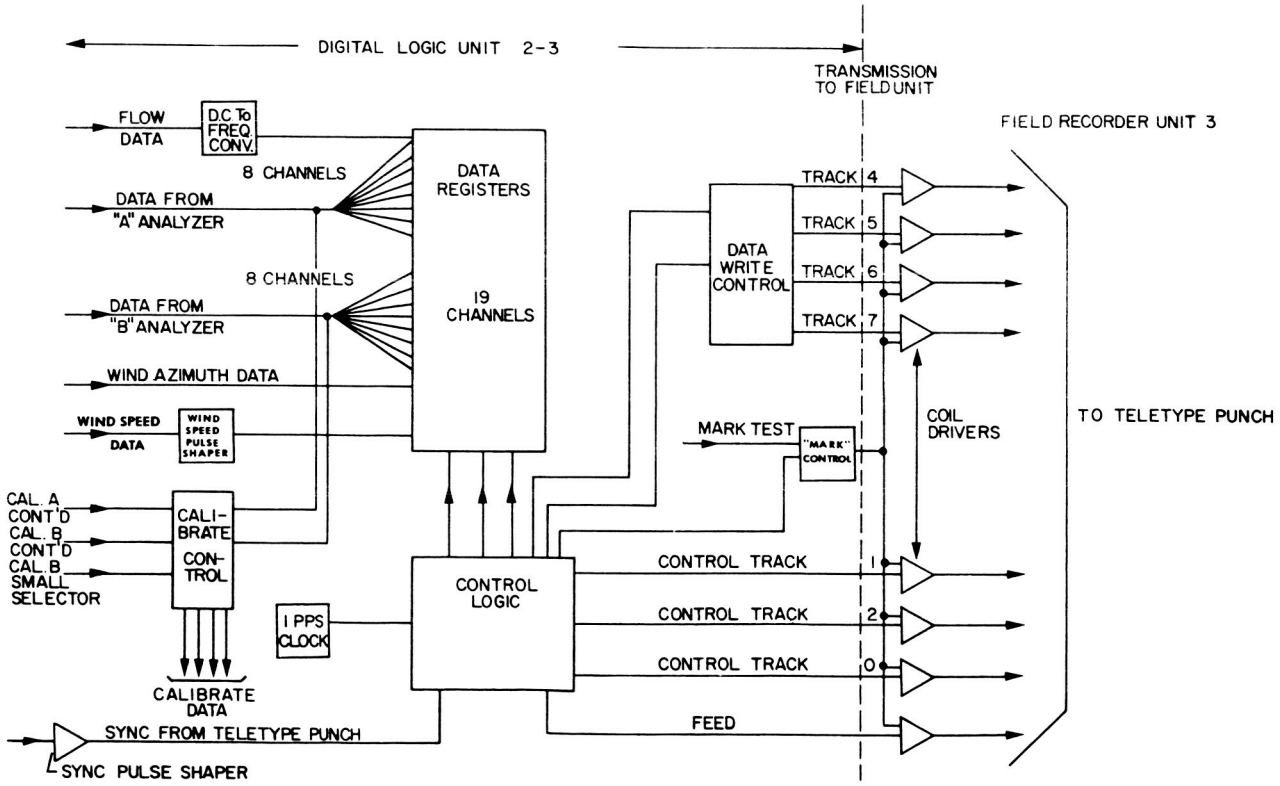



Figure 19. Block diagram of field digital logic Unit 2-3

recorded indicate the particle count categorized according to particle size in each of the eight size categories from each sampler. It also receives and records wind speed and direction from the meteorological sampler and the volumetric sampling rate from Sampler A. In addition a test mark time is indicated.

Particle count data are derived from the photomultiplier output pulse height analyzers on each unit. Measurement of a particle results in a signal to one of a set of binary counters each with a capacity of 10 binary digits. The system accumulates counts in each of the 16 size categories for a 1 second period. At the end of this time, the totals are entered on the printer tape punch and the counters are reset to zero; counting then resumes for the next 1 second block. The tape, therefore, contains a succession of blocks with binary numbers containing the particle count data, wind speed, direction, and volumetric sampling rate over 1 second observation periods.

With this particular system, individual tests of duration up to approximately one hour are considered. At the end of a test, the roll of paper tape is taken from the field electronics and hand carried to the information center (see Figure 20). The information center then reads the tape and transfers the recorded data into the storage unit. From time to time, as indicated by settings on the programmer unit, playback is halted and accumulated data is printed via a high speed printer. The main components of this information center are the playback unit, the program control and decoding unit, the accumulated data storage system, and the data printout system. The playback unit is a standard teletype paper tape readout system which feeds information from the data blocks into the program control and decoding unit.

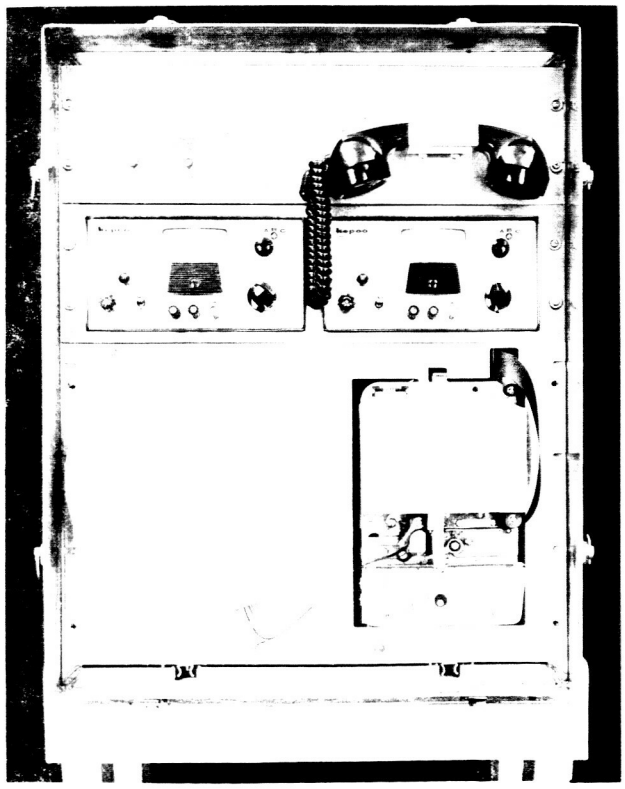
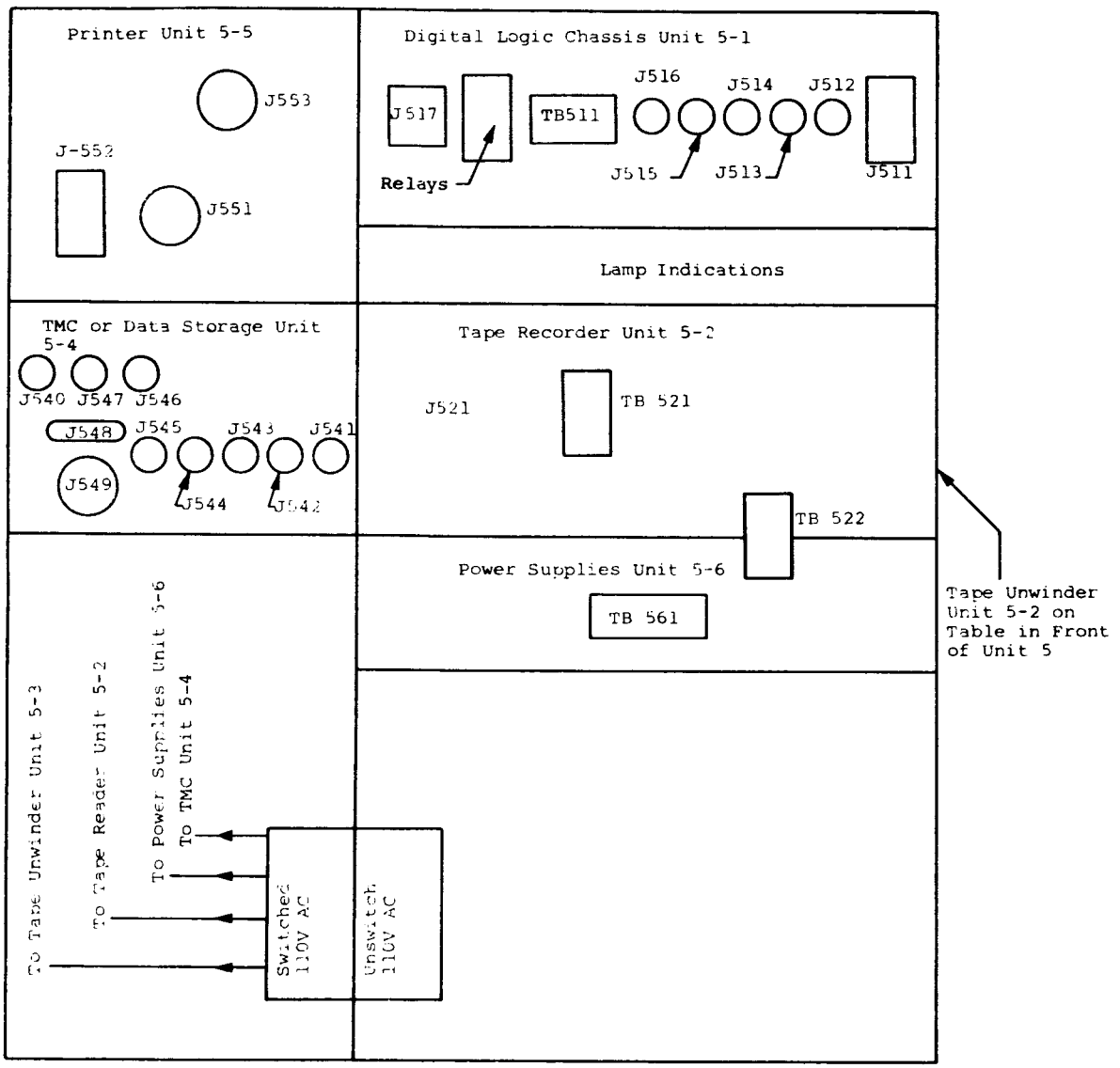


Figure 20.
Field recording Unit 3

The program control and decoding unit has the following functions. First, a decoder recognizes the time marks which signal the beginning of individual blocks of data. A preset program selector permits readout to be limited in terms of either time or flow. The programmer reads out data in multiples of 1 second blocks and transfers the data to the appropriate channel and data storage system, in terms of particle size, wind speed or time.

The data storage unit is a 100 channel counter with additional provisions for readin and readout and display. This unit receives pairs of numbers from the playback unit and decoder, stores, accumulates, and transfers data in accordance with preprogrammed instructions to the printout unit. The printout unit is a high speed Munro printer with a capacity of 17 lines per second. Upon command from the programmer output, accumulation is halted at the end of a specified block of information and printout begins. The printer successively interrogates each channel of the data storage unit and prints out two numbers, the channel number and the total count accumulated in that channel. Readout is destructive so that the data storage is cleared for the next set of data blocks and readout of the recorded data can continue.

The complete unit operation is shown in block diagram in Figure 21. This unit was designed for testing of aerosol generation and transmission systems for a specific purpose. Its applicability in terms of capability for remote operation and automatic control would make it ideal for collection and storage of data in remote areas or in areas where personnel costs are greater than equipment costs. Obviously the construction and development costs of this prototype unit are high, probably beyond the capability of a single industrial laboratory or municipality. However, the lower replication costs should make it of interest to such groups. The great capacity for data handling and multiple point sampling should make it ideal for computer-oriented, remote operating systems.



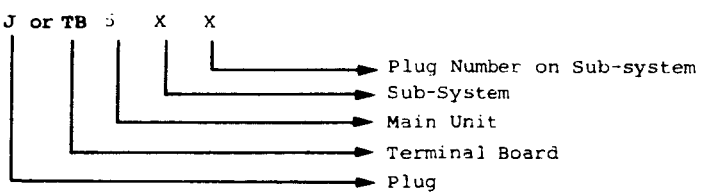




**Figure 21. Data reduction center function block layout and connector location**

# 3. MONITORING PARTICLES BELOW 0.3 MICRON PARTICLE SIZE USING THE CONDENSATION NUCLEI COUNTER, AND THE APPLICATION OF THIS INSTRUMENT TO HIGH EFFICIENCY FILTER LEAK TESTING

by

G. E. HELMKE

Bell Telephone Laboratories, Incorporated
Murray Hill, New Jersey

## Abstract

Particles of the 0.001 micron to 0.1 micron radius size range are called condensation nuclei because of the technique used in their detection. In this technique the submicroscopic particles to be detected serve as nucleation sites on which water droplets form and grow to a size readily detected by light scattering methods. A condensation nuclei counter may be used to detect and locate leaks in HEPA (High Efficiency Particulate Air) filters and in gaskets used in connection with their installation in laminar flow clean rooms and clean work stations. This leak detection method takes advantage of the fact that condensation nuclei, which are present in great numbers in ordinary room air, are largely retained by a HEPA filter. By scanning the downstream side of the HEPA filter with a continuously operating condensation nuclei counter, the sections of the filter, its support frame, and gaskets, which fail to remove nuclei can be readily delineated. Used in this way, the condensation nuclei counter serves as a reliable and accurate tool for testing for leaks in HEPA filter installations.

# 4. A METHOD FOR DETECTING LOW CONCENTRATIONS OF AIRBORNE GASEOUS CONTAMINANTS

by

R. A. YEICH

Western Electric Company
Reading, Pennsylvania

Abstract

The General Electric Condensation Nuclei Counter, with suitable converters, may be used to monitor concentrations of sulfur dioxide and certain hydrocarbons. This paper indicates problems encountered and items of value discovered in using this equipment.

The Reading Plant of the Western Electric Company has been manufacturing solid state devices since 1953. In 1957 WE installed its first clean room (See Figure 1). At the present time, there are facilities of all classes as listed in Federal Standard No. 209, from clean rooms and clean benches to controlled ambient systems. Instrumentation used to monitor and detect particles includes a Royco Photometer, Royco Particle Counters (both 200 and 220 Models), Dynac Model 101 Particle Counters and the General Electric Condensation Nuclei Counter. One method of detecting and monitoring low concentrations of airborne gaseous contaminants is with the nuclei counter and converters.

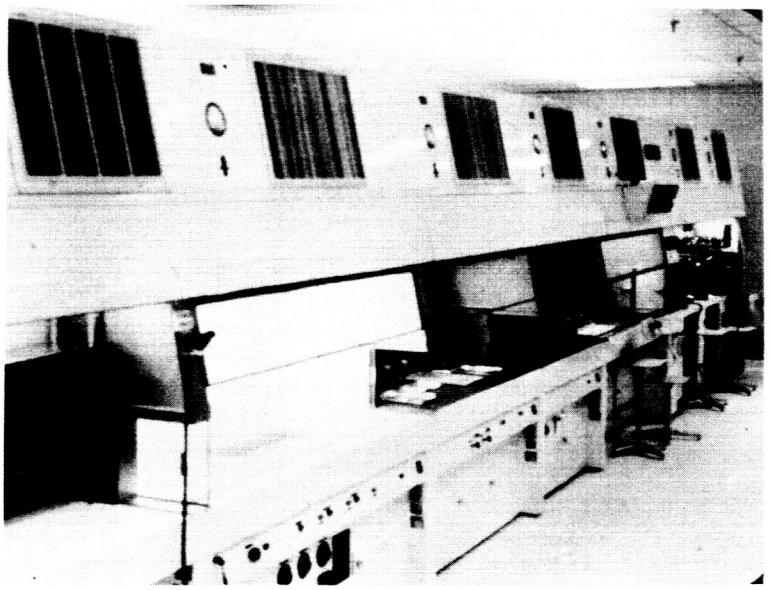


Figure 1. Clean room at Reading Plant at Western Electric

The condensation nuclei counter is not actually a counter, but a photometer which measures the forward scattered light in a dark-field optical system (see Figure 2). This instrument is unique because it enables monitoring of particles in the submicroscopic range of 0.001 micron to 0.1 micron. By use of a pump and suitable valving, the gas sample to be examined is passed into a saturating chamber, where the relative humidity is raised to 100%. The sample is then passed into a drywall expansion chamber containing the dark-field optics. After the inlet valve closes, a valve opens into a chamber which has a negative pressure of 8 inches of mercury to allow adiabatic expansion of the sample. This expansion is extremely rapid and causes the relative humidity to rise from 100% to above 400%. Condensation is initiated on particles present in the sample in about one millisecond. A 0.001 micron droplet of water will grow to 5 microns in about 26 milliseconds, a size suitable for optical detection. If condensation is initiated on all particles in the sample at the same time, the rapid growth will negate any difference in particle size, and the light scattered will be proportional to the number of particles in the sample.

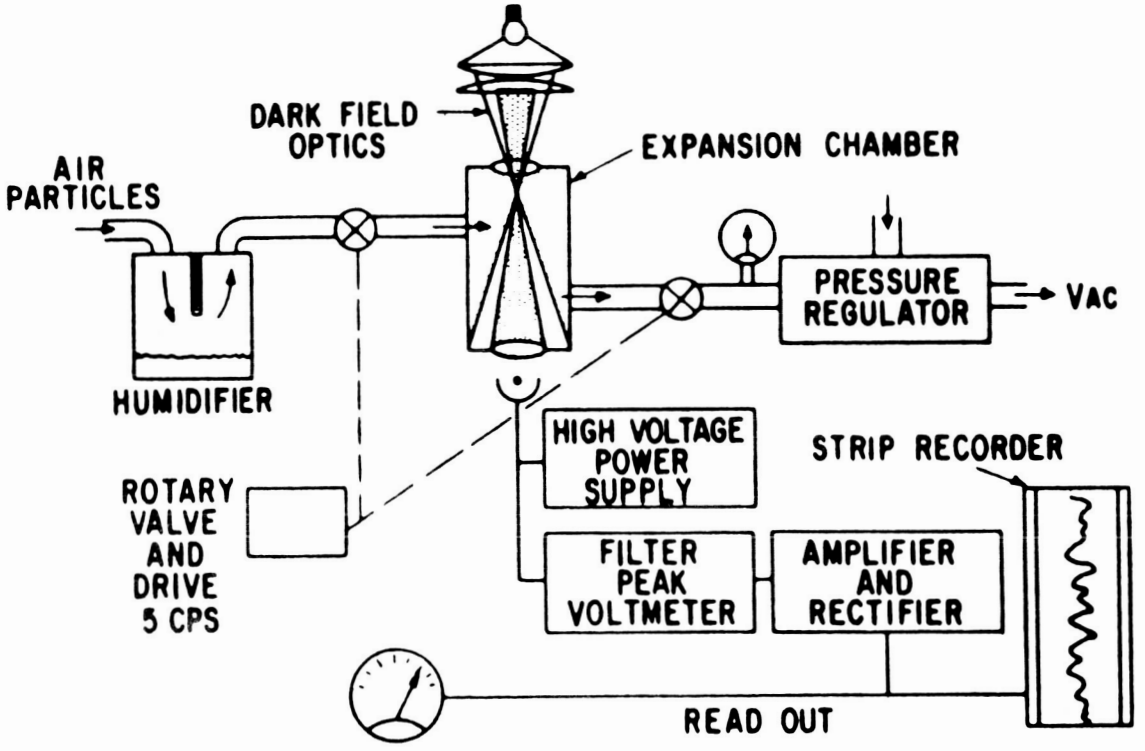



Figure 2. Condensation nuclei detector

The intensity of the scattered light is read by a photomultiplier tube and, with suitable circuitry, displayed on a meter (see Figure 3). The meter is divided into linear steps of 300, 1,000, 3,000, 10,000 and 100,000 nuclei per cc and exponential steps of 1,000,000 and 10,000,000 nuclei per cc. The sequence of operation is completed 5 times per second, thereby permitting a response time of only two seconds from intake to readout of a sample.

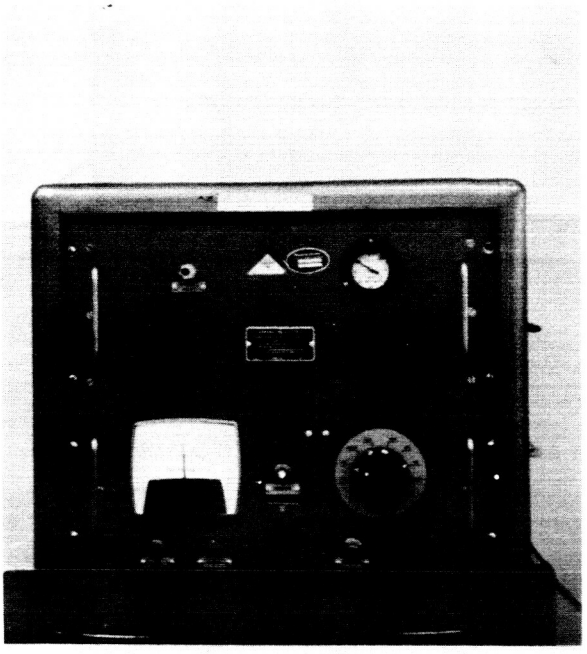
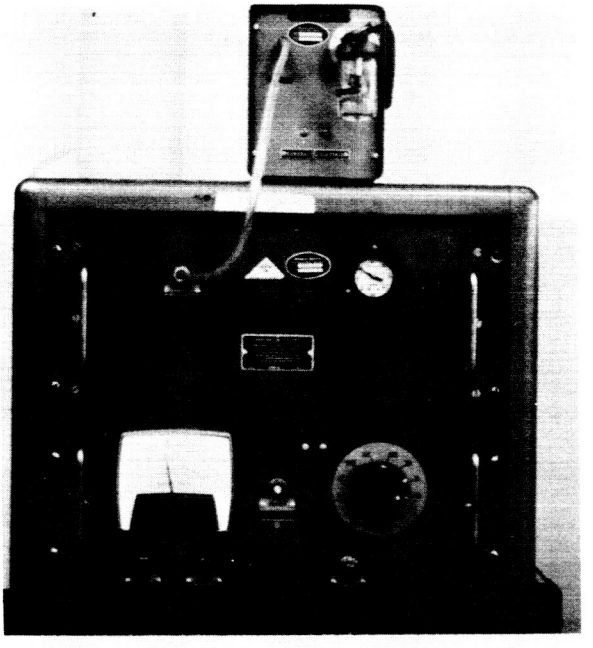


Figure 3.
Meter display of intensity of scattered light

Under operating conditions, the valving accuracy is rated at ±20% above 0.3 full scale on linear ranges. Connections are provided on the back of the nuclei counter to attach leads to a strip chart recorder. Difficulty has been experienced in using the nuclei counter as a monitor when it is coupled with the converters, because of the extreme sensitivity of the tests.

One of the gas converters is the sulfur dioxide converter (see Figure 4). This extremely simple item is composed of an ultraviolet lamp, constant voltage transformer, off-on switch, Gelman filter, and

Figure 4.
Sulfur dioxide converter



a reaction flask which contains the ultraviolet lamp--all housed in a small metal cabinet.

The gas sample through tygon tubing, is drawn through the sulfur dioxide converter and into the condensation nuclei counter. The sample gas first passes through the Gelman filter, which is sized to remove 99.7% DOP-0.3 micron and larger. In other words, the attempt is to remove most of the particulate matter which would scatter light without condensation. The sample gas is then passed through the ultraviolet chamber, where free oxygen produced by a photochemical reaction unites with sulfur dioxide to produce sulfur trioxide. The sample then passes into the condensation nuclei counter, where the sulfur trioxide unites with the water of the saturating chamber to form droplets of sulfuric acid. These droplets then become the nuclei upon which condensation begins in the dark-field optical chamber. The meter readings of the condensation nuclei counter are converted by a chart, which is supplied with the converter, to parts per million sulfur dioxide (see Figure 5). The sensitivity range is from 0.004 parts per million to 0.035 parts per million (ppm) on the linear scales and from 0.035 parts per million to 0.2 parts per million on the exponential scale.

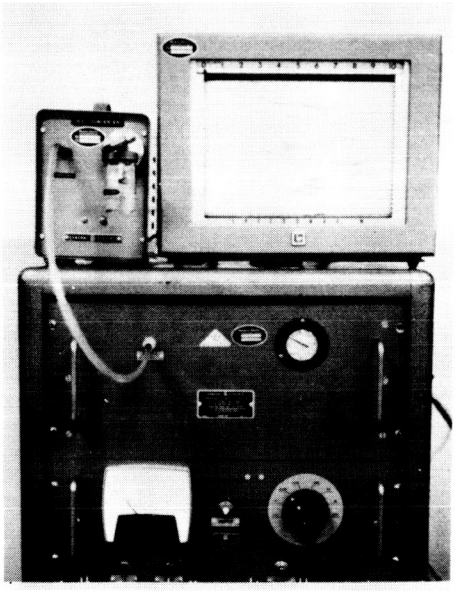


Figure 5.

Chart conversion of meter reading

If an operator is in constant attendance and the meter on the nuclei counter is used, we have no problems. However, if we wish to use the nuclei counter as a monitoring instrument, we must attach a strip chart recorder (see Figure 6). The linear scales from 300 to 100,000 are easily readable on the chart. The exponential scales start at approximately 32% of the linear scale and read to maximum, which makes interpolation on a strip chart rather difficult. We are, therefore, inclined to use only the linear scales (see Figure 7). The 100,000 scale at full scale reading is equivalent to 0.035 ppm sulfur dioxide. It is disconcerting to arrive at the monitoring station late in the afternoon and to discover that, from 10 a.m. to 4 p.m., the instrument has been off scale and we have no idea how high the level of contamination has been (see Figure 8). Frequently the contamination level rises and falls sharply. Projection of the curve would indicate astronomical levels and, obviously, would be completely erroneous.

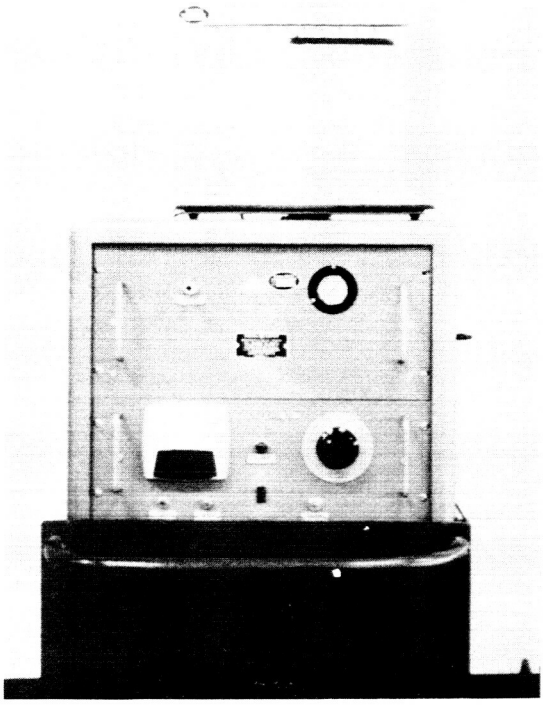


Figure 6.
Strip chart recorder

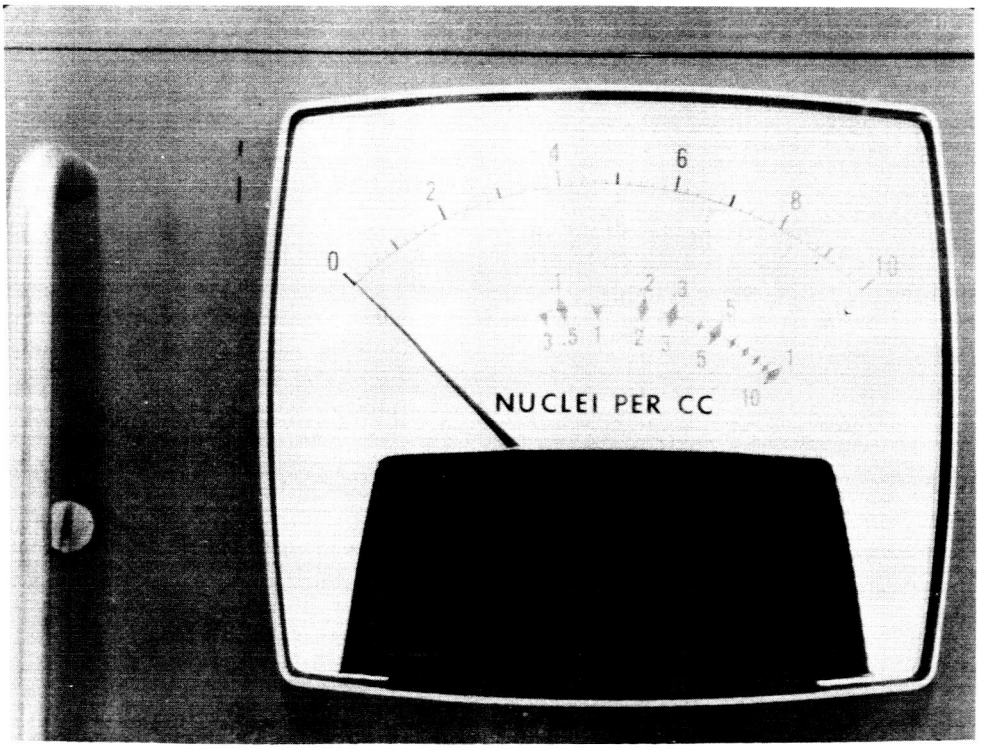



Figure 7. Linear scale (100,000 full scale)

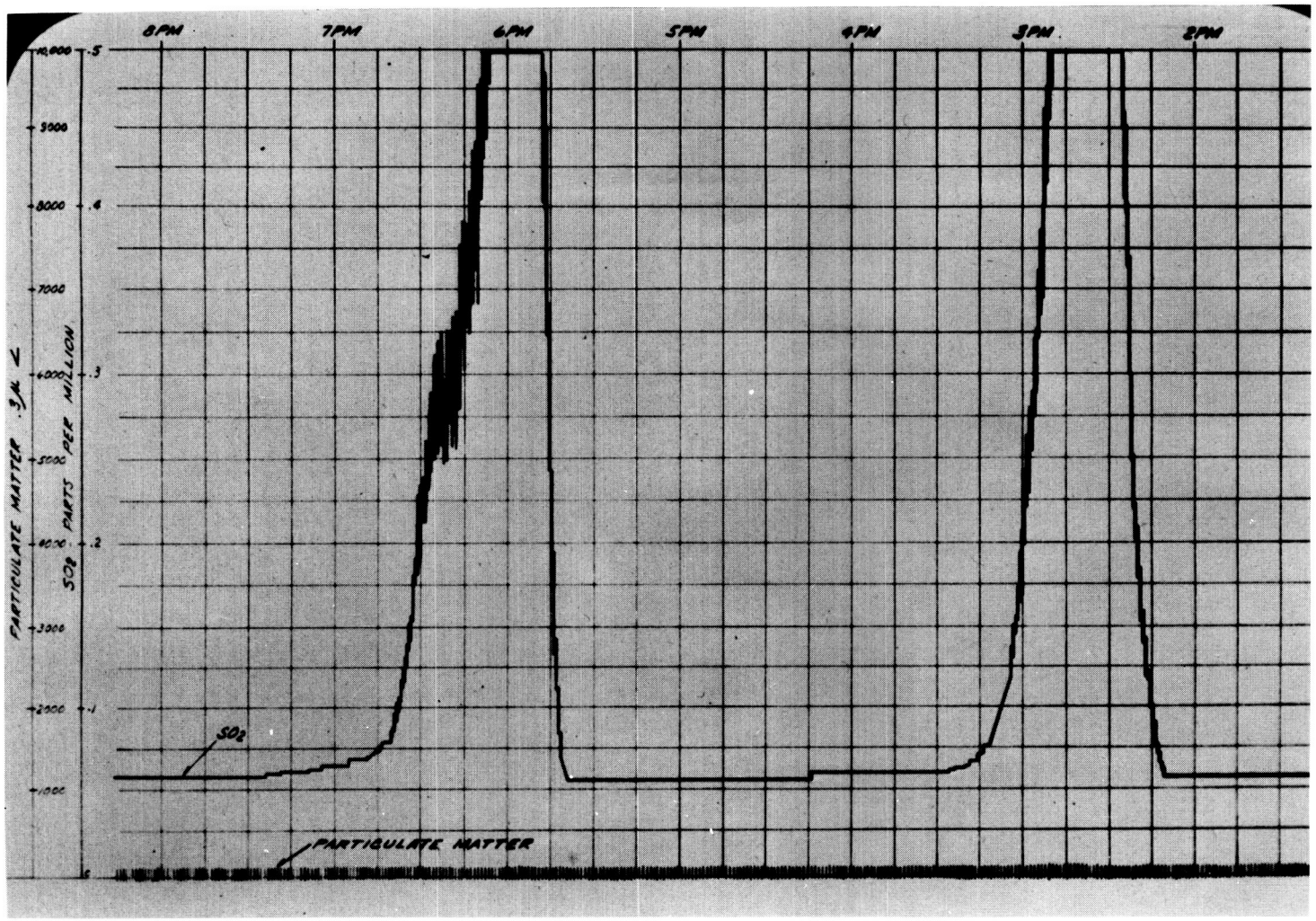



Figure 8. $SO_2$ count in a clean bench

Monitoring on the 10 million scale provides a range from 0.06 to 0.2 ppm sulfur dioxide, and the one million scale provides a range from 0.02 to 0.12 ppm sulfur dioxide. Both of these ranges have the attendant difficulty of interpolating the results of the exponential reading on a linear chart.

Exponential charts can probably be procured, but this would cause additional expense, and added problems when there is switching to a lower range because charts would then have to be changed. These are not insurmountable difficulties, but they do bear mentioning.

The hydrocarbon converter is similar to the sulfur dioxide converter in internal construction (see Figure 9). The difference is that sulfur dioxide gas is introduced in the gas stream after it leaves the reaction flask containing the mercury vapor lamp. The amount of sulfur dioxide is minute and difficult to adjust.

An additional problem encountered in the unit tested is that all the leaks in the system cannot be eliminated, and sulfur dioxide is released to the ambient. This leakage contaminates the ambient and causes extreme discomfort to personnel working in the area.

The General Electric instruction manual does not indicate the chemical reaction which takes place. Dr. Douglas Bird of General Electric indicates that a possible olefin detection mechanism is generated when complex hydrocarbon molecules are exposed to ultraviolet light in the 2537 Å range (see Figure 10). The molecules may form radicals and, in the presence of sulfur dioxide, recombine to form larger molecules, which could be a liquid and could also be more water attractive since they contain sulfur dioxide. These would permit detection by the nuclei counter.

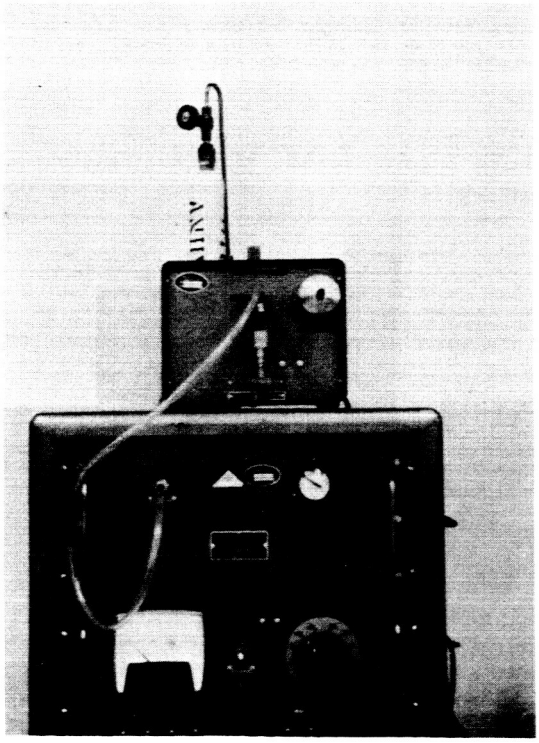


Figure 9.
Hydrocarbon converter

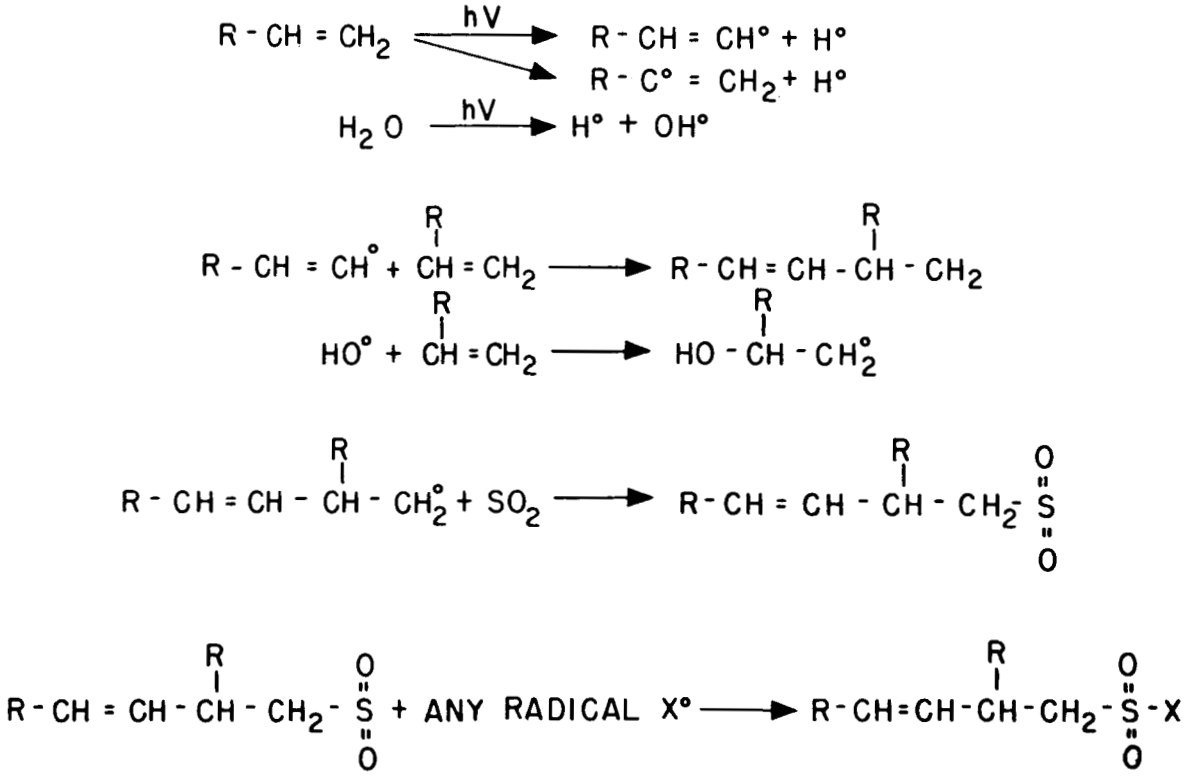


Figure 10. Possible chemical reaction

(Ref. 1. - J. G. Calvert & J. N. Pits Jr. "Photo Chemistry" pp 200 & 502 to 505
2. - R. D. Snow & F. E. Frey "Ind. Eng. Chem." 30, 176, 1938)

It was observed that alcohol vapors inhibit the reaction in the hydrocarbon converter. No explanation was given by General Electric, but this factor may limit the usefulness of this instrument and should be taken into account if the monitor ceases to function in an area where alcohol is in use. Most lighter fluid vapors will cause a full scale indication on the high range and can be used to check operation of the hydrocarbon converter.

The manual indicates that the hydrocarbon calibration is based on octene ($C_8H_{16}$) vapors. Limited testing in the WE plant did not indicate a change in hydrocarbon level when samples of ambient were taken in the vicinity of a vacuum pump which has visible vapors emanating from the exhaust port while it was running. However, we did detect a rise in hydrocarbon contamination during the shift changes, which we tentatively attribute to automobile traffic.

All of the problems mentioned previously with the sulfur dioxide converter and with monitoring with a strip chart recorder, apply here to a greater extent. The meter range of the nuclei counter and hydrocarbon converter is in the 100,000 to 10,000,000 condensation muclei per cc range; therefore, all readings are made within the exponential scales. The range is from 2 to 20 parts per million octene. It must be admitted that WE has not investigated the hydrocarbon converter as fully as the sulfur dioxide converter, but its potentialities appear to be limited in its usage at the Reading Plant.

A major problem encountered with the nuclei counter is the lack of a simple method of testing for accuracy and repeatability. A particle counter which operates above 0.3 micron is easily tested with an aerosol solution. The nuclei counter can only be checked by voltage readings and oscilloscope graphs; however, we found this to be of little concern in using this equipment at Reading. We feel the readings obtained are relative, not strictly quantitative, and the extreme sensitivity of the instrument reduces the problem of accurate calibration to a minimum in the ranges which are significant to our processes. Functioning of the gas converters can easily be checked with a match. If a match is ignited, promptly extinguished and then held at the sample intake orifice a full scale indication on the highest range will be noted with either converter.

The Reading Plant of the Western Electric Company manufactures semiconductors and high frequency power tubes. The semiconductor work encompasses epitaxial, diffusion and thin film work in transistors, diodes, and integrated circuits. All of this work requires clean conditions in one phase or another. The present technology does not permit the removal of vapors and fumes in either clean benches or clean rooms, and knowledge of vapor conditions is useful.

The value of the nuclei counter with the sulfur dioxide converter may be indicated in the following example. A problem was encountered with thin aluminum stripes evaporated onto germanium slices (see Figure 11). Sulfur dioxide in the ambient air was suspected. The slices in question were stored, after deposition of the aluminum stripe, in dishes in holding ovens (see Figure 12). The dishes were ceramic baking dishes with removable covers. Pure nitrogen gas was introduced into the dishes by a copper tube through holes in both the oven and dish. Sulfur dioxide was detected in the dish under normal operating conditions, and readings fluctuated with the readings in the ambient. Sulfur dioxide was not eliminated until every opening in the oven was sealed, including the joints in the metal skin and even the opening around the toggle switch. The only input was pure nitrogen gas, and the only outlet was the sample tube with "tee," which conducted the gas from the oven to the sulfur dioxide converter and exhausted the excess gas to the ambient, preventing back flow into the oven.

When one considers clean room applications, we recommend that the nuclei counter be used outside of clean rooms wherever possible. The cooling air is discharged from the side of the equipment without filtration, and we have experienced problems when monitoring in the same room with a standard particle counter. Counts are higher when the cooling air discharge from the nuclei counter are directed over the intake tube of the particle counter. When it is used in a clean room, the counter should be thoroughly cleaned and the instrument positioned so that the air discharge is directed away from sensitive processes.

The nuclei counter and sulfur dioxide converter are extremely useful for detecting minute quantities of sulfur dioxide in the ambient air. They are sensitive and reliable, as can be verified by our two years of almost constant monitoring. The value as a quantitative instrument is limited because of a lack of ease in checking operation and the narrow range of contamination measured on the linear scales and the limitations of readout and sensitivity on the exponential scales. The simplicity of operation far outweighs the faults of readout in the investigative area.

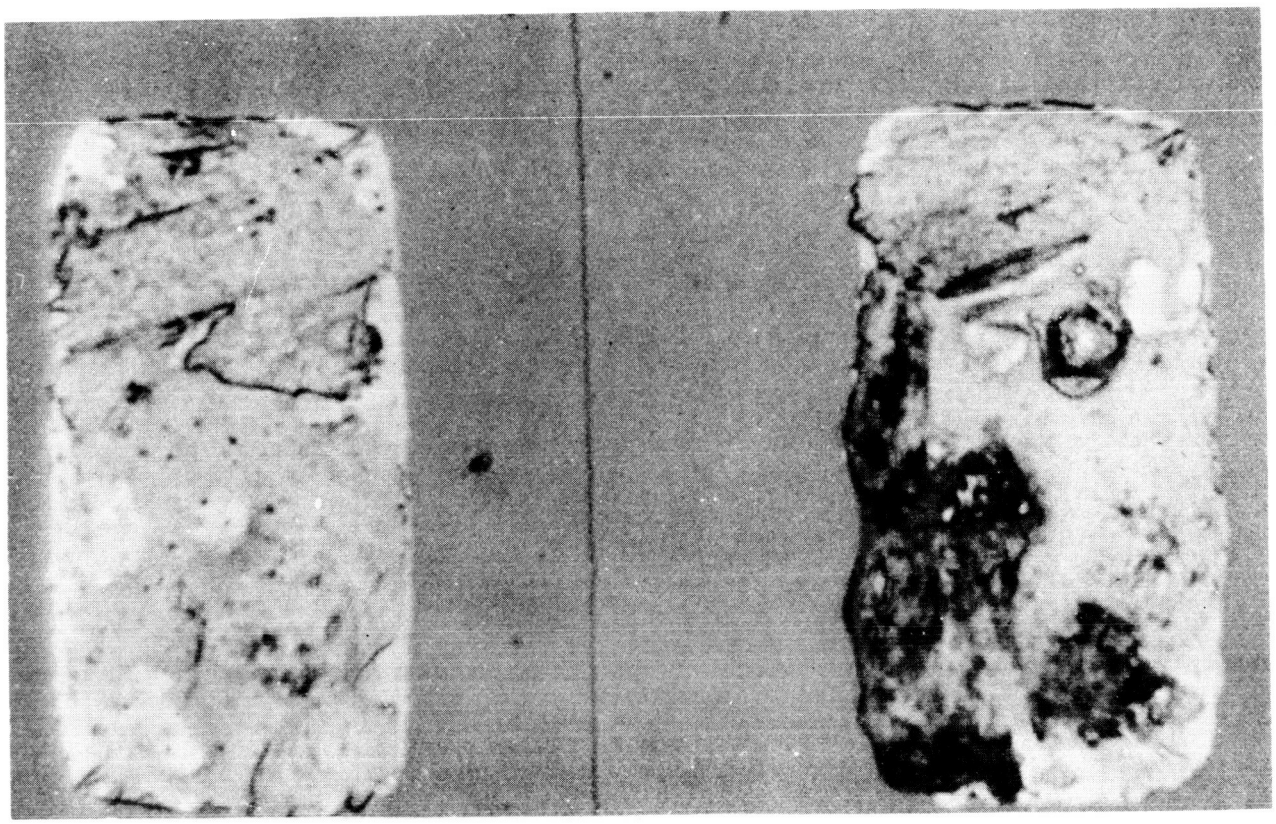


Figure 11. Aluminum stripes evaporated onto germanium slices

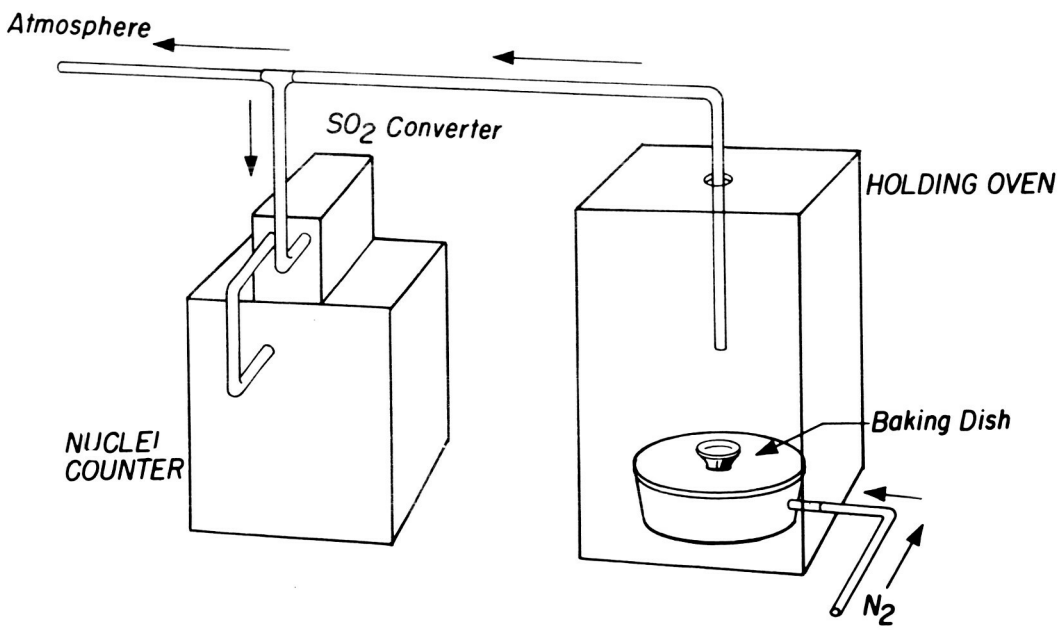



Figure 12. Equipment for work with slices

The hydrocarbon converter is difficult to use because of the extremely irritating nature of the activating gas and the limitations of readout. We have experienced difficulties with leaks in the converter, which limit its usefulness to areas where exhaust hoods are available. The exhaust from the nuclei counter itself must be vented to a suitable exhaust system because it contains sulfur dioxide and is quite corrosive. We feel that the nuclei counter and sulfur dioxide converter are valuable additions to WE's clean room instrumentation when the limitations are recognized and they are used within the areas of their capabilities.

Specifications for the equipment were obtained from the instruction manual supplied by General Electric. Assistance by E. L. Johnson and Dr. Douglas Bird of the General Electric Company Research and Development Center of Schenectady, New York, and Mr. Roy D. Derr, of the Western Electric Company, are gratefully acknowledged.

# 5. A STUDY OF HEPA FILTER EFFICIENCY IN SUBMICRON PARTICLE RANGE

by

L. J. Klamerus
and
M. I. Tillery,
Lovelace Foundation

Presented by

L. J. KLAMERUS

Manufacturing Research Division
Sandia Corporation
Albuquerque, New Mexico

Abstract

This report describes various techniques used to evaluate the efficiency of HEPA filters on particles less than 0.3 micron in size. A brief discussion is given of a gas sampling procedure used in a laminar flow clean room. Efficiency figures are given for HEPA filters on Uranine aerosol particles less than 0.3 micron in size.

## Introduction

The collection efficiency of High Efficiency Particulate Air (HEPA) filters for submicron particles is of more than academic interest. People using these filters, whether for laminar flow clean rooms, clean benches, mine safety, or reactor safety, have much information on their efficiency for particles larger than 0.3 micron in size. Light-scattering instruments designed for monitoring and testing these filters have a lower limit of 0.3 micron; thus, filter manufacturers specify the efficiency of the filters in terms of particles greater than 0.3 micron in size.

Viruses, in the 0.003 to 0.06 micron range, and harmful vapors are examples of undesirable constituents of some air which can pass through the filters.

Briefly, the problems involved with testing the efficiency HEPA filters in removing these small particles are:

1. To generate large quantities of very small particles.
2. To determine the size and number of these particles.
3. To detect the small amount of penetrating mass.

***

This work was supported by the United States Atomic Energy Commission.

Various techniques used to evaluate the efficiency of the filters are described in this paper. All testing techniques used isokinetic sampling, and the air velocity downstream of the filter was between 95 and 100 feet per minute.

## DOP Challenge

The technique of using a hot or cold dioctyl phtholate (DOP) smoke generator to test HEPA filters is well known. The equipment generally used is a DOP smoke generator, DOP liquid, blower, plenum, a short tunnel, clamps, and some light-scattering instrument capable of giving a reading on particles 0.3 micron in size.

A concentration of smoke is introduced on the upstream side of the filter or filter bank to be tested. The downstream side of the filter or filter bank is scanned completely with the probe of the particle sensor. The test is usually "go-no-go." A reading above some prescribed level results in either repair or replacement of the tested filter.

This method is probably the most universally used and does perform its intended function of testing the filters for leaks on particles 0.3 micron in size. Copious quantities are fogged out of the generator and in many instances, although little is known of the exact size distribution, the output is called 0.3 micron in size. Actually, an appreciable percentage of the massive total quantity of particles is near enough to this size to give a leak signal.

Since the particles generated are dependent in number and size on several parameters which are usually unknown, there must be some doubt about the actual particle size. The condensation of the hot DOP requires nuclei to be present, and the number and size of these nuclei vary with experimental arrangement. Also, temperature and dilution rate are factors which affect the size distribution. In the cold DOP generator, the nozzle and impactor designs and operating parameters are all important and can be controlled; yet, there is still some spread in the distribution for a single generator and more variance between different generators.

To repeat, the usual method of generating DOP for filter testing does a job. It was not adequate for Sandia purposes in checking efficiency on particles less than 0.3 micron in size, since particle size distribution is uncertain, and the practical limit for light-scattering instruments on small numbers of particles is 0.3 micron.

## Gas Sampler

The suspected presence of harmful vapors within a laminar flow clean room of one of Sandia's suppliers prompted an investigation. This provided an opportunity to extend the effort to determine the efficiencies of HEPA filters on particles below 0.3 micron in size, since molecules of the gases entering the room are two orders of magnitude smaller than some particles generated and observed during other phases of this evaluation.

Several two-liter flasks with two pressure stopcocks were fabricated. Spherical flasks were used for ease of cleaning. These flasks were evacuated and taken to the supplier's plant, where gas samples

were taken inside the clean room and outside of the plant. Upon return to Sandia, the contents were analyzed on a gas chromatograph and mass spectrometer and by chemical analysis.

The chemical analysis used was as follows:

Ozone - liberation of $I_2$ and titration sodium thiosulfate with a starch indicator.

Nitrogen Dioxide - direct absorption in a colorimetric reagent.

Sulfur Dioxide - formaldehyde colorimetric method on alkaline absorbing solution.

A sensitivity of 0.01 PPM was obtained in this analysis, and periodic monitoring will be maintained.

## Cesium Chloride and Sodium Chloride Aerosol

In this series of tests, solutions of cesium chloride and sodium chloride of 1%, 0.1%, and 0.01% concentrations were aerosolized. The generator used was a miniature high-output aerosol generator as of the type described by Newton, Bennick, and Posner.[1] It aerosolized at a rate of 3 ml/hour.

These aerosols were driven into the plenum chamber of a test tunnel, and attempts were made to capture them both above and below the HEPA filter. A thermal precipitator (Cassela) and an Electrostatic Precipitator (see Figure 1) were used to capture particles on standard carbon-film electron microscope grids. The Electrostatic Precipitator is described by Mercer, Tillery, and Flores,[2] and is unique since it uses a tritium source to ionize the particle and a strong electrostatic field to precipitate the charged particle.

Electron micrographs were taken of the grids on a Phillips EM 75 electron microscope, and the particles counted and sized with a Zeiss Particle Counter. A computer program was used to arrive at statistical data on the distribution. The particle size varies as the cube root of the concentration, but even in the 0.01% concentration solution with Count Median Diameter (CMD) of 0.24 micron, the particles captured were too few to give meaningful statistics.

The method revealed that the filters were very efficient on particles 0.1 micron and larger in size, and also showed the inadequacy of the slow generator rate and collection rate. The thermal precipitator sampled at 8 cc/min and the Electrostatic Precipitator at 9cc/min.

## Silver Chloride Furnace Generator

An induction heated furnace was used to achieve a temperature of 650°C to vaporize Silver Chloride. Condensation nuclei were formed and were driven with dry nitrogen into the plenum of the test tunnel.

---

[1]Page 29 - LF-33 - Biol. & Med. TID-4500; Lovelace Foundation for Medical Education and Research

[2]LF-7 - Instruments TID-4500; Lovelace Foundation for Medical Education and Research.

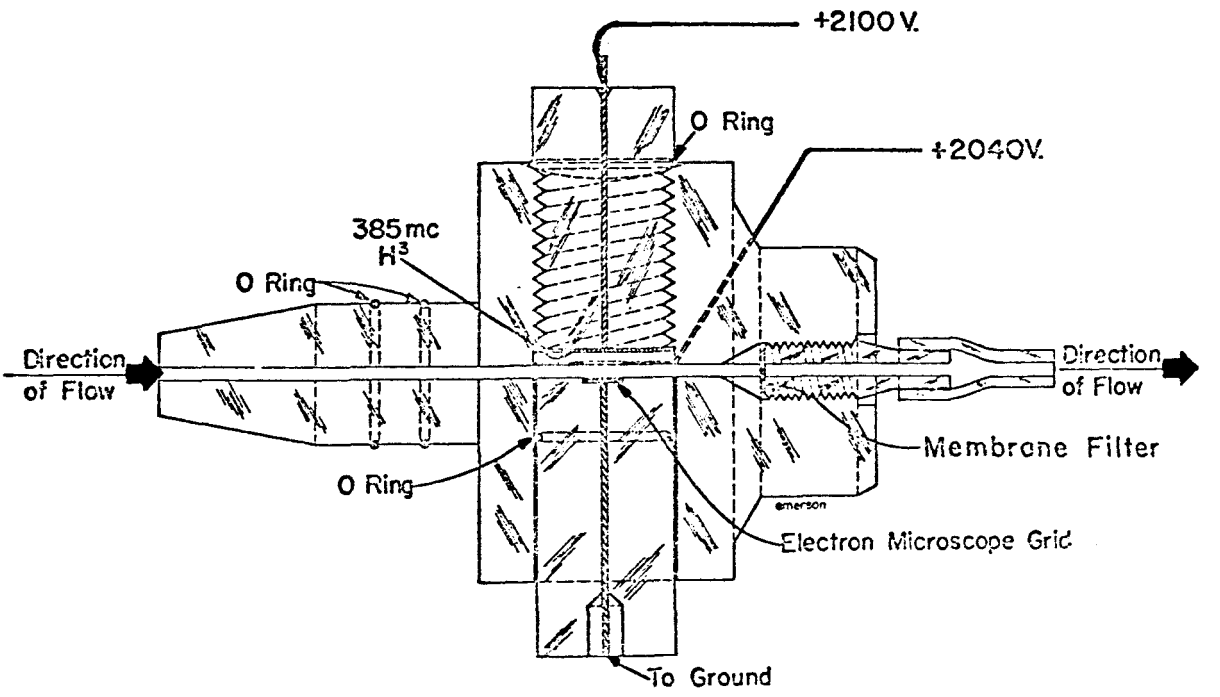



Figure 1. Diagram of electrostatic precipitator

The electrostatic precipitator, electron microscope, and data reduction techniques were used as described. A marked reduction in particle size was achieved; the Mass Median Diameter (MMD) of several runs was 0.033 micron, and the Count Median Diameter (CMD) was 0.020 micron with a geometric standard deviation $\sigma_G$ of 1.31. The particle size was not reduced enough to increase the mass present below the filter to the point where sound statistical information could be obtained.

## Irradiated Sodium Fluoride Aerosol

The limited success of the previous techniques indicated a need for an easily detectable small particle. A radioactively tagged particle seemed the answer. Irradiated sodium fluoride was chosen as a readily water soluble powder with a fairly large cross-section and relatively short half-life isotope. Sodium 24 has a half-life of 15 hours.

It was decided to use the miniature aerosol generator described previously and a seven-stage impactor upstream and also downstream of the HEPA filter to capture the particles. The fourteen stages were then put into a Beckman Wide Beta Counter to find the activity on each stage. The upstream impactor operated at 150 cc/min and the downstream impactor at 1 liter/min.

Two tests were run. The first test used 0.6 milligram of NaF irradiated to an activity of 1 millicurie and dissolved in 6 ml. of water, giving a 0.01 percent concentration. The particles produced had an aerodynamic mass mean diameter (AMMD) of 0.64 micron, an MMD of 0.38 micron, and $\sigma_G$ of 1.68 as observed on the upstream impactor. Of the total mass generated, 20 percent consisted of particles less than 0.4$\mu$ aerodynamic mass diameter (AMD) or 0.24$\mu$ mass diameter (MD).

The second test used 3 milligrams of NaF irradiated to an activity of 8 millicuries and dissolved in 30 ml of water, giving a 0.01% concentration. However, a different generator was used; particles were produced having an aerodynamic mass mean diameter of 0.36 micron, a MMD of 0.22 micron, and a $\sigma_G$ of 1.5. Of the total mass generated, 32.5% was less than 0.3$\mu$ AMD or 0.18$\mu$ MD. The generator used was an ultrasonic device which aerosolized at a rate of 30 ml per hour.

The data from both tests fit a log-normal distribution quite well (see Figure 2). This plot[3] is typical of those obtained for aerosols. Enough information was obtained, once again, on upstream distribution, but the efficiency of the filters did not allow enough particles to pass downstream to permit information retrieval. It had been hoped to obtain a plot similar to that shown in Figure 2 for the downstream impactor. Ideally, this plot would be smaller and squeezed to the lower diameter particles, but with the upstream plot would provide a point-by-point comparison of efficiency. The activity measured in both tests was only two to three times background, which was too low to rely upon.

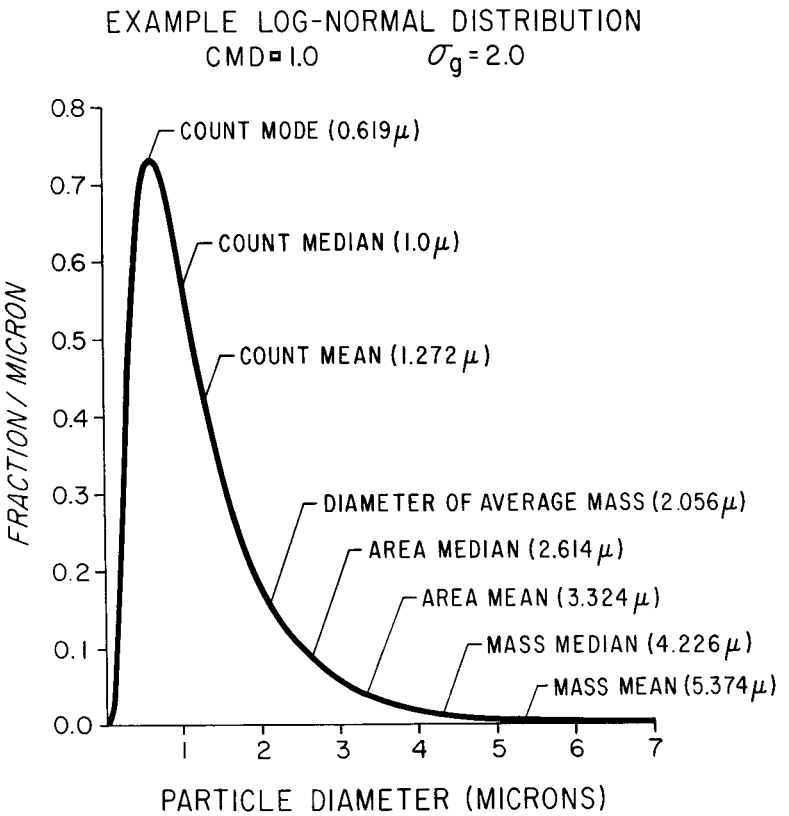



Figure 2. Plot from two tests with miniature aerosol generator

[3]Unpublished paper, "Particle Size Analysis Utilizing Grouped Data and the Log-Normal Distribution" by Otto G. Raabe, PhD, Fission Products Inhalation Laboratory, Lovelace Foundation.

Uranine Solution Aerosol

The previous tests showed the high efficiency of the filters was preventing sufficient mass from arriving downstream. The tests did show, however, that some small particles were getting through. A massive attack was planned whereby a continuous test would be run for several hours by using the ultrasonic generator for maximum output and providing, by sheer volume, enough mass downstream of the filter.

The length of time involved ruled out the use of a radioactive isotope. Instead, sodium fluorescein (uranine) was chosen for its fluorescent properties, and fluorometer was used to measure the fluorescence. The seven-stage impactor with a flow rate of 920 cc/min was chosen for the upstream detector, and a membrane filter backed by activated charcoal was operated at 51.7 liters/min downstream.

Tests were run for 5-3/4 and 6-1/2 hours. The ultrasonic generator aerosolized the 0.01% uranine solution at a rate of 30 ml per hour. A total of 36.7 mg of sodium fluorescein was generated in this manner. Twenty-five cu ft of air per minute flowed through the tunnel during all tests, providing a velocity of 100 feet per minute downstream of the filter.

Good correspondence between the test data was obtained. On Test One, the particles had an AMMD of 0.51 micron, an MMD of 0.44$\mu$, and a $\sigma_G$ of 2.2. Of the mass generated, 30 percent was of particles less than 0.35 micron AMD or 0.30$\mu$ MD. Test Two produced particles with an AMMD of 0.44 micron, an MMD of 0.38$\mu$, and a $\sigma_G$ of 2.01. Of the mass generated, 30 percent was of particles less than 0.30 micron AMD or 0.26$\mu$ MD. Figure 3 shows the distribution of the uranine solution upstream of the HEPA filter in Test Two.

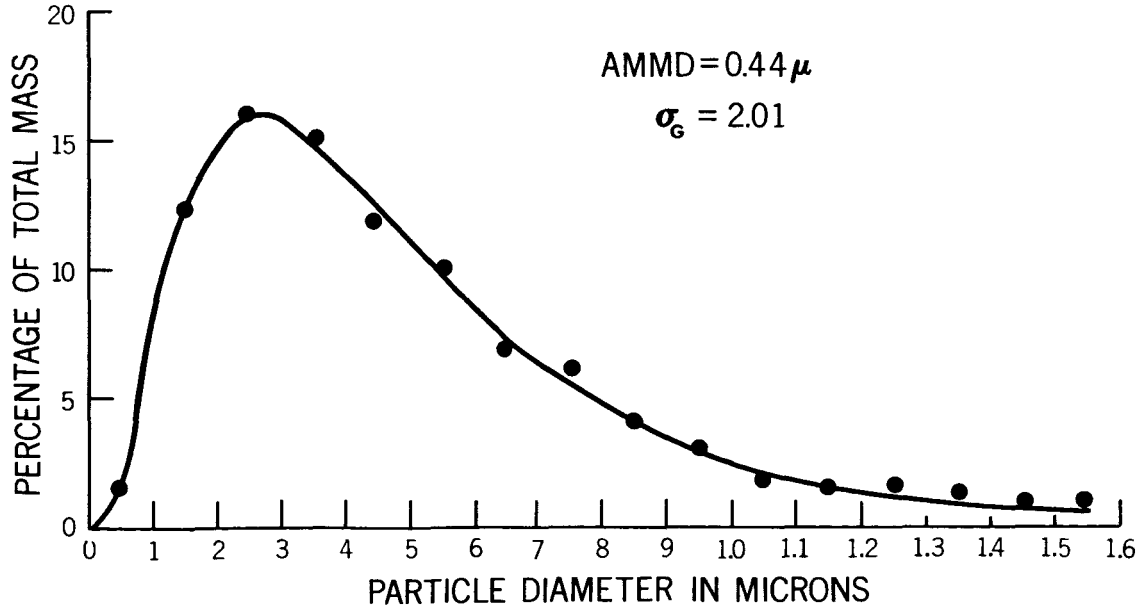



Figure 3. Mass and size distribution of uranine aerosol upstream of HEPA filter

The density obtained on the inpactor upstream was $8.88 \times 10^{-6}$ mg per liter, while the density found downstream was $3.56 \times 10^{-9}$ mg per liter. This would indicate an efficiency of 99.96% over all sizes generated here. If it is assumed that all particles above 0.3 micron are filtered out successfully, as previous electron micrographs indicate, the efficiency is 99.87% on the 2.66 mg per liter of all particles less than 0.3 micron.

## Conclusion

All test techniques used indicated the HEPA filter to be well within the manufacturer's specification of 99.97% efficiency on all particles 0.3 micron in size. For the particular size distribution generated with the uranine solutions, the filter tested 99.87% efficient for those particles less than 0.3 micron in size. This high efficiency makes most test techniques suffer from insensitivity.

Further efforts will be made to find where the cutoff exists on these filters, as one must exist somewhere above a diameter of an air molecule. Various gases will be generated upstream and detectors placed downstream to intercept particles, if any pass through the filters. This test is necessary to evaluate the effect of contaminated air entering a clean room and contaminating objects and personnel. Additional techniques which may be used are a Condensation Nuclei Counter and a Diffusion Battery. Both are sensitive to very small particles, but have low sampling rates.

BURLING: Will low humidity environments (about 10% RH at 68°F) have an effect on particle monitoring in Class 100 clean rooms?

LIEBERMAN: The answer to that is yes. Most humidity systems will have an effect on particle monitoring. This involves the entire system, which includes the sampling network as well as the particle analysis device. As far as the particle analysis device is concerned, humidity will probably have little if any effect. The optic light scatter systems, for example, are seldom, if ever, affected by humidity. Electrical monitoring systems are affected by humidity since the conductivity of the atmosphere and of the environment will be modified by relative humidity. The problem is that when the humidity goes down so does the efficiency of the sampling system itself. The reason is that many of the particles get a barometric constant; at low humidity they will pick up an electric charge. If they pick up an electric charge and if the electric field and the atmosphere is such that there is a voltage difference between the sample and the particle then the chances are present that the particle may deposit right at the sample inlet and not get into the sampling system at all. This is the most serious problem to be considered in low humidity environments.

BALLARD: Does ASTM F-1 recognize the DOP challenge as an acceptable alternate to use of condensation nuclei for testing HEPA filters?

HELMKE: Yes. The ASTM F-1 committee now has in preparation a standard for the use of DOP challenge as a filter testing method. I am not recommending the condensation nuclei counter technique as a substitute or replacement for DOP. Each has its own specific area of use. For work on components or devices in which the cleaning agent is freon, trichloroethylene, alcohol or other solvents, the DOP test is fine. For electronic devices, where substrates are cleaned with boiling hydrogen peroxide solutions, we look for the absence of hydrocrobic contaminants on the substrate and thus we try to avoid the recontamination that may occur in the short period after DOP.

GILBERT: Let me amplify a bit. There has been a filter challenge standard completed by the United States Standards Institute and it is in the process of approval. The American Association of Contamination Control (AACC) is aware of this standard and will probably adopt it. This standard will be cross-referenced probably in ASTM F-1.

BALLARD: Does the high efficiency of HEPA filters for intercepting condensation nuclei (0.001 micron in size) mean that virus interception should be equally as effective?

HELMKE: I believe 1/10 micron might be a fair size for virus, and if this is so, I would assume, although not certain, that the HEPA filter would intercept a virus.

KLAMERUS: In general, the size of the virus goes down from 1/10 micron. That is one of the things we were looking for in our evaluation. The filters were very effective down to 1/10 micron range, but this does not negate a window which possibly may exist for certain sizes, and, in that case, viruses of that size could proceed through the filter.

GOULET: If all particles become 10 micron in size, how do you know how many particles of different sizes are present?

HELMKE: With the condensation nuclei technique, there is no way to size particles. All that can be said is that the particles counted are in this range.

GOULET: What is the sample flow rate of the nuclei counter?

HELMKE: The expansion chamber of the nuclei counter draws in 20 cc's five times per second. This comes to 100 cc/sec or 6 liters/min.

GOULET: How long does it take to scan a filter?

HELMKE: For a five foot bench, a filter could be scanned in about 5 minutes.

CADWELL: Is the size of a water droplet dependent on the size of the nucleus about which the droplet formed?

HELMKE: To the best of my knowledge, it is not. Particle physicists who have studied this matter believe that they are looking at particles in the droplets in the 10-micron range.

STEINBERG: Since the forward light scattering photometer works on mass of particles, how can you say that it will not detect under 0.3 micron?

KLAMERUS: It is a function of the number we are talking about. There is no doubt that a forward scanning photometer will pick up large masses of smaller particles. I contend that light scattering devices cannot pick up molecules because we are proceeding so far below the wave length of light which established this lower limit, essentially around 3/10 micron. But with large masses of any size, scattering will be picked up.

STEINBERG: The hot DOP generator carefully controlled will produce greater than 99 percent of 0.3 micron particles. You state differently. Do you have other information?

KLAMERUS: The key words here are "carefully controlled." The hot DOP generator, as I said, is a function of four different parameters, which, unfortunately, many people do not control.

GILBERT: We were able to get particles from 1/10 micron to 1 micron out of the nozzle of the cold DOP generator. With the hot DOP generator (the fixed nonportable filter testing DOP penetrometer), the particle size is very carefully controlled by a device which holds the particle size at 3/10 micron.

BEYERLE: It became apparent early in the aerospace contamination control program that the methods of testing fluids were time consuming, were costly and not always reproducible. Many thousands of fluid samples were previously tested in typical aerospace cleaning operations to verify and meet the stringent demands imposed by our specifications reliably. Automatic counters appeared the most promising to control these requirements. We at Marshall Space Flight Center, however, were not in the business of developing instrumentation for monitoring. So we had to depend upon industry and research institutes for the R and D phase. We were interested only in the end product development and also

in a stringent reliability program to assure meeting the requirements for the clean hardware. I would like Mr. Lieberman to discuss the ramifications of this R and D phase.

LIEBERMAN: This was a coordinated operation. Mr. Beyerle had a problem and IIT ended up as the place where a solution was sought. In essence, IIT got from Marshall Space Flight Center a set of requirements. The requirements were not to develop a particulate or automatic particle counter to certain design specifications, but for IIT to come up with instrument parameters of particle size limitation, particle size concentration measurement capabilities, data handling and things such as these. In short, what we had to do was to get together with Marshall and develop a process for an instrument specification. From there we were to go to an instrument design. From the large number of complex slides that have been shown here the last couple of days, all should agree that a major part of the initial research and development work is completed.

Instruments have been designed to meet essentially all of the requirements. The performance has been specified, and the performance is based on the hardware capabilities available in present day devices, such as photomultipliers, relays, transistors, valves, motors and so forth. We know that we need to monitor cleanliness and we know what the requirements of clean room operation are. What is also implicit in all of these instrument performance requirements is the designability, and this is where the going gets tough. The instrument design has been done, there are about a half dozen available commercial instruments for monitoring airborne particles, there are available another half dozen or so instruments for monitoring particles in liquid, and there are about another 20 designs available for laboratory devices--one of a kind prototype systems. This is at the point where we are now--reliability and operation.

GILBERT: This is a step in the right direction. In the AEC, reliability is something we have to require. We have field conditions and the filter banks were not designed and installed for testing. The testing came later, and it gets rather rugged to get the instruments up to the filter bank for the testing. Consequently we prefer a field type instrument. Obviously it should be as portable as possible because there are some rather difficult conditions under which field people have to test. It is simply a case that we would like the technician doing this work to be as happy as possible, but with the instrumentation that we have, while we made some progress, there is much left to be desired. Does anyone want to amplify the situation as to what the future might offer, where we might do more work, and whether the cost of instrumentation and techniques might come down? Has anyone volunteered to support the de-bugging of the IIT instrument?

LIEBERMAN: As far as anybody volunteering money to support the de-bugging of the IIT instrument, no, this will not happen. Eventually, either industry or government will find that they require reliability of instruments which may or may not be an instrument designed by the IIT. It may be an instrument designed by any of the manufacturers that may require modification for special purpose, high reliability, or detail operations. At this time, there is no money available for such needed de-bugging of new designs.

GILBERT: What is an approximate figure for the cost of nuclei counters and the attachments for the chemical monitoring?

YEICH: The base instrument costs about $4500 and each one of the attachments runs approximately $500. Subsequent to writing my paper, I received a letter from General Electric which indicated that for another $1500 it could provide an automatic range changing mechanism in the equipment so if one were on one range and passed out of that range into a higher range, the mechanism would automatically go into the next higher range. GE also indicated if any sulphur dioxide molecules are present in the ultraviolet chamber, it can, by masking them, permit different ranges to be set up. Neither of these things was I familiar with when I wrote my paper.

HELMKE: I was much intrigued by Mr. Klamerus's paper, and it is important for all of us to know the efficiency of HEPA filters. I hope that we will soon be able to have someone provide a detailed account of these filters. I would suggest, Mr. Klamerus, that you look at the possibility of generating particles from electric arcs of the size needed since Sandia seems to have the facilities for checking sizes.

KLAMERUS: That is a helpful suggestion. Now, I would like to pose a question. What should be done during the gas sampling techniques if smog enters the room? Charcoal filters or water shower?

YEICH: At Reading, we checked into charcoal filters several years ago, and, in my opinion, they are useless. One day, I was doing a test and discovered that I was releasing more sulphur dioxide from my filter than I was putting into it. We found that somebody was over in the corner using a spray gun. It appeared that the charcoal filter had more affinity for the solvents in the spray gun, so it was getting rid of the sulphur dioxide.

GILBERT: I would disagree with you. I think the size of your sample must have been inadequate. We have had good success with charcoal filters in our area. We have investigated many substitutes for charcoal, and we still find it to be the best for our application, which is primarily the absorption of iodine.

KLAMERUS: I was not speaking of small areas. I was thinking of charcoal filter banks to absorb smog in large amounts.

ELINSKY: A requisite is to know what contamination levels are being experienced in the gaseous phase, which is a different contaminant than the particulate of clean rooms. Carbon filters have a fixed capacity for absorption. Therefore, Mr. Yeich was experiencing the overabundance of the concentration which had been picked up and was coming through the downstream side.

SESSION V

MICROBIAL CONTAMINATION

Presentations

| | | |
|---|---|---|
| 1. | MICROBIAL DECONTAMINATION AND SAMPLING PROGRAM FOR ANCHORED INTERPLANETARY MONITORING PLATFORM (AIMP-E) SPACECRAFT | -- F. N. LeDoux |
| 2. | ASSAY TECHNIQUES FOR PLANETARY QUARANTINE | -- M. S. Favero |
| 3. | THE VACUUM PROBE FOR REMOVING ORGANISMS FOR COUNTING | -- M. E. Morris |
| 4. | THE PROBABILITY OF RELEASING OF MICRO-ORGANISMS ON FRACTURE FROM SOLIDS | -- M. J. Peterson |
| 5. | LIFE DETECTION EXPERIMENTS | -- G. L. Hobby |

Chairman

A. H. NEILL
NASA Headquarters
Washington, D. C.

SESSION V

MICROBIAL CONTAMINATION

# INTRODUCTION

by

A. H. NEILL

NASA Headquarters
Washington, D. C.

The Program Arrangement Committee has done an outstanding job of selection. The program is varied, it is down to earth, and it also gets into the so-called never-never land.

We, in the Planetary Quarantine program, are faced with formidable problems in trying to implement some of the NASA policies and objectives in maintaining the pristine state of the planet and also in containing the contamination on the Lunar surface, as well as keeping an inventory of its location and type of contamination so that in the future, if we do have successful explorations, we will know where that contamination is so that it will not confuse personnel in their very vital search for extraterrestial life. That is the near program objective.

We have approached the planetary quarantine functions in a fundamental manner. In the past few years, there has been a substantial supporting research and technology development program through the planetary quarantine bioscience program. There are about 30 projects underway at the present time searching for better ways to do the job and to make it more and more compatible with the engineering design, assembly, and other mission objectives. The biologists have learned to recognize that they cannot sit off on a little island by themselves and do the job. It has got to be done within the framework and understanding of the engineers, technicians, designers, and everyone else.

This seems to be one of the major problems even now--to get a good understanding among the viable contamination people, the nonviable contamination people, the engineers, and anyone else that has to do with the testing, assembly, and so on. I think that this kind of meeting tends to promote this understanding.

# 1. MICROBIAL DECONTAMINATION AND SAMPLING PROGRAM FOR ANCHORED INTERPLANETARY MONITORING PLATFORM (AIMP-E) SPACECRAFT

by

F. N. LeDOUX

Goddard Space Flight Center
Greenbelt, Maryland

Abstract

A requirement of the Office of Planetary Quarantine, NASA Headquarters for biologically clean spacecraft operating in the near vicinity of the moon necessitated the development of a decontamination program for the AIMP-E spacecraft.

Decontamination was effected with chemical solutions of isopropyl alcohol and acetone. To determine effectiveness of decontamination process two methods of recovering viable micro-organisms were used. One method employed control strips with detachable coupons for monitoring the electronic circuit modules and the other method employed sterile swabs and templates to monitor other surface areas. Coupons and/or swabs were immersed in a 1% peptone wash solution and sonicated at 25 kc/sec for 12 minutes. Aliquots of the sonicated solution were plated out on agar, incubated and colony counts made. Records were made of the numbers of aerobic and anaerobic spore and vegetative organisms remaining on a surface after the decontamination process. All assembly and test operations were conducted in controlled and/or clean-room facilities. As a result of the evaluation of records that were maintained the AIMP-E spacecraft contained an internal burden of $2.19 \times 10^5$ organisms and a surface burden of $4.42 \times 10^4$. The surface spore loading was estimated at $7.4 \times 10^3$ organisms. This spore population on the surface will be reduced to less than $1.89 \times 10^{-9}$ due to the spacecraft's orbital life expectancy of 3 years and 1440 cycles of temperature change in an ultra high vacuum.

## Introduction

A primary objective of the Anchored Interplanetary Monitoring Platform (AIMP-E) is to investigate the characteristics of the interplanetary magnetic field out to and at lunar distances in either a captured lunar orbit or a geocentric orbit with an apogee near or beyond the lunar distance. Because of the spacecraft's mission in the near vicinity of the moon it is considered a potential lunar lander as the gravitational pull of the moon will eventually capture the spacecraft and it will impact the lunar surface. When this occurs a number of viable micro-organisms remaining on the spacecraft carried from the earth will be deposited on the lunar surface.

It being a prime responsibility of the NASA Bioscience program to record and maintain inventories of all biological contamination deposited on the lunar surface it then became necessary for the Goddard Space

Flight Center (GSFC) to device an in-house program for biological decontamination; sampling and assaying the spacecraft and its hardware, maintaining a bio-clean environment during the phases of assembly and testing and a method of maintaining running records of viable contamination.

Before a program for biological decontamination of the AIMP-E spacecraft was inaugurated tests were conducted to determine the compatibility of the spacecraft's materials and the decontaminates used to reduce the microbiological population. Samples of all materials used in the build-up of the spacecraft system were tested, i.e., all metals coated and bare, fiberglass, epoxies, sealants, adhesives, thermal coatings, plastics and working electronic components. As a result of all the tests conducted there was no evidence to substantiate that failure of a component would occur as a result of the decontamination process.

All surfaces of the spacecraft were monitored for microbiological contamination. The various areas were classified with respect to the manner in which they were exposed or occluded and as to their physical location. Figure 1 depicts the AIMP-E spacecraft in a flight configuration.

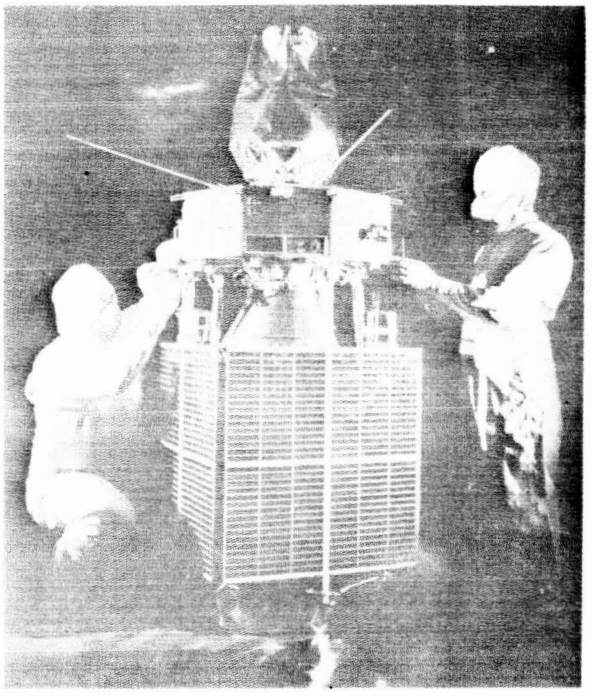


Figure 1.
AIMP-E spacecraft in a flight configuration

Area Classification

A. Interior surfaces prior to occlusion by conformal coating and/or encapsulation. All interior surface areas of the module frames which included walls, cavities, and electronic circuit boards.

B. Occluded surface areas obstructed by module frames excluding exposed surfaces of module frame stacks.

C. Cover and occluded inner surfaces of the spacecraft.

D. Other interior surfaces of the spacecraft.

E. Exterior surfaces of the spacecraft occluded and exposed.

Methods Employed for Microbial Recovery

Two methods of recovering viable organisms from the spacecraft surfaces were employed. One method employed control strips with detachable coupons. Figure 2 shows a control strip in detail. Figure 3 shows such a control strip affixed to a circuit module frame. The control stips were first sterilized in a steam autoclave then affixed to

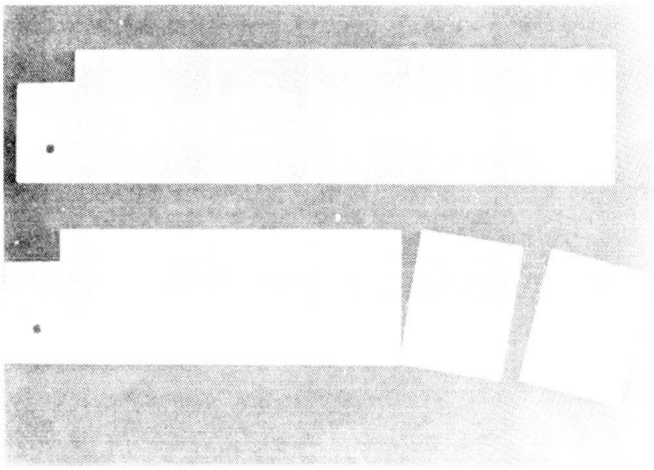


Figure 2.
Control strip with detachable coupons

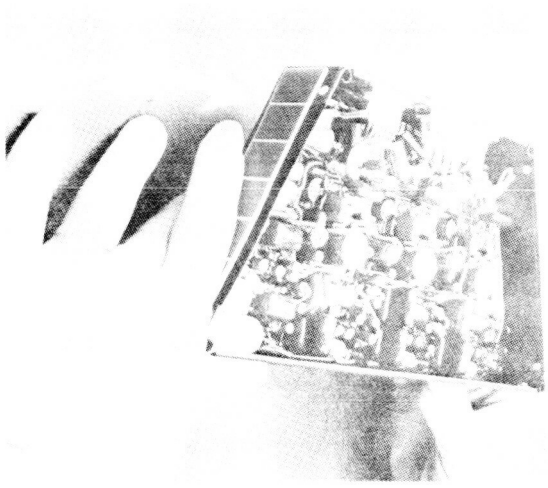


Figure 3.
Electronic circuit module with control strip attached

each module frame in a similar manner, so located as to compel the technician to touch the control strip each time the module is handled. The purpose of the control strip is to prove the effectiveness of decontamination process and allow a practical method of assaying the contamination level on a circuit module prior to decontamination and the probable level of contamination after the decontamination process. These control strips were fabricated from the same material as the printed circuit board and in such a manner as to yield five easily removed coupons.

A coupon was removed from the control strip and assayed to determine the contamination level. After the decontamination process another coupon was removed so as to determine the number of viable organisms remaining on the circuit module. Upon removing a coupon it was placed into a wash bottle containing 50 ml of 1.0% sterile peptone solution. The wash bottle was then placed in an ultrasonic bath and sonicated at 25 kc/sec for 12 minutes before petri-plate preparation.

The circuit modules were monitored biologically in this manner at time of conformal coating and again at time of encapsulation.

The second method of recovering viable micro-organisms employed sterile swabs and templates. Sampling was accomplished by first inserting the sterile swab into the wash solution. The swab was then retracted into its holder and the excess solution removed by pressing the swab tip on the inside wall of the tube. A rolling twisting motion was used. Sterile Kraft paper was used as a template. The template openings varied depending upon the configurations of areas sampled. In most cases a 4 square inch opening template was used. After sampling a particular area the microbiologist performing the sampling held the swab by its protective shield and aseptically broke it off into its test tube which contained 5 ml of sterile 1.0% peptone wash solution. The test tube containing the swab was then placed into an ultrasonic bath and sonicated at 25 kc/sec for 12 minutes. This procedure dispensed the cotton into the peptone solution. Figure 4 shows the type of swab used to obtain the samples from the spacecraft and its hardware.

Petri-Plate Preparation. Eight, 100 mm-diameter petri plates were prepared from each coupon wash bottle. Four, (4) plates were prepared each containing 5 ml of the sonicated solution and 20 ml of trypticase soy agar (TSA). The remaining sonicated solution was heat-shocked at 80°C in a water bath for 20 minutes. Four, (4) petri plates were prepared each containing 5 ml of the heat-shocked solution. Four, (4) petri plates were prepared from each swab sample. Two, (2) plates were prepared each containing 1 ml of solution and 20 ml of TSA. Remaining solution was heat-shocked at 80°C for twenty minutes. Two, (2) plates were then prepared each containing 1 ml of the heat-shocked solution.

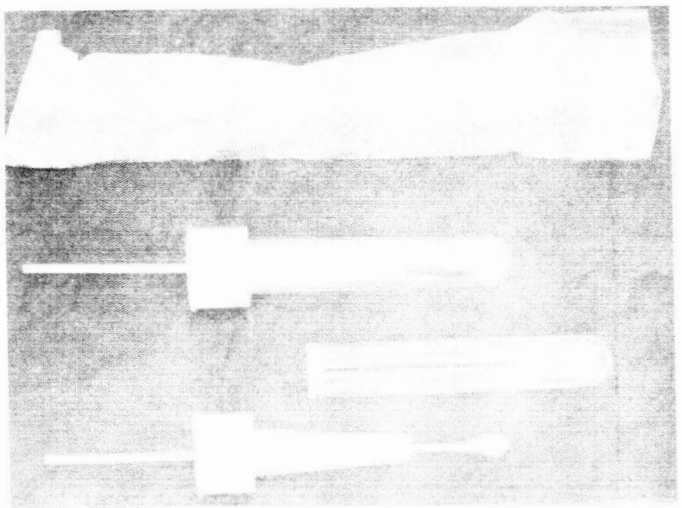


Figure 4. Sterile swabs used for sampling areas for assays

Culturing. Two (2) petri plates prepared from the nonheat shocked solution obtained from the coupons and two, (2) plates prepared from the heat-shocked portion of this solution were aerobically incubated. Colony counts were made after 24, 48, and 72 hours. Remaining plates containing nonheat-shocked and heat-shocked portions of solution were anaerobically incubated. Colony counts were made after 72 hours of incubation. Culturing of the solution obtained from the swab samples were performed in the same manner. All petri plates were incubated at 32°C. Culturing normally was started within an hour after taking samples from the spacecraft. Figures 5a and 5b depict the manner of culture preparation.

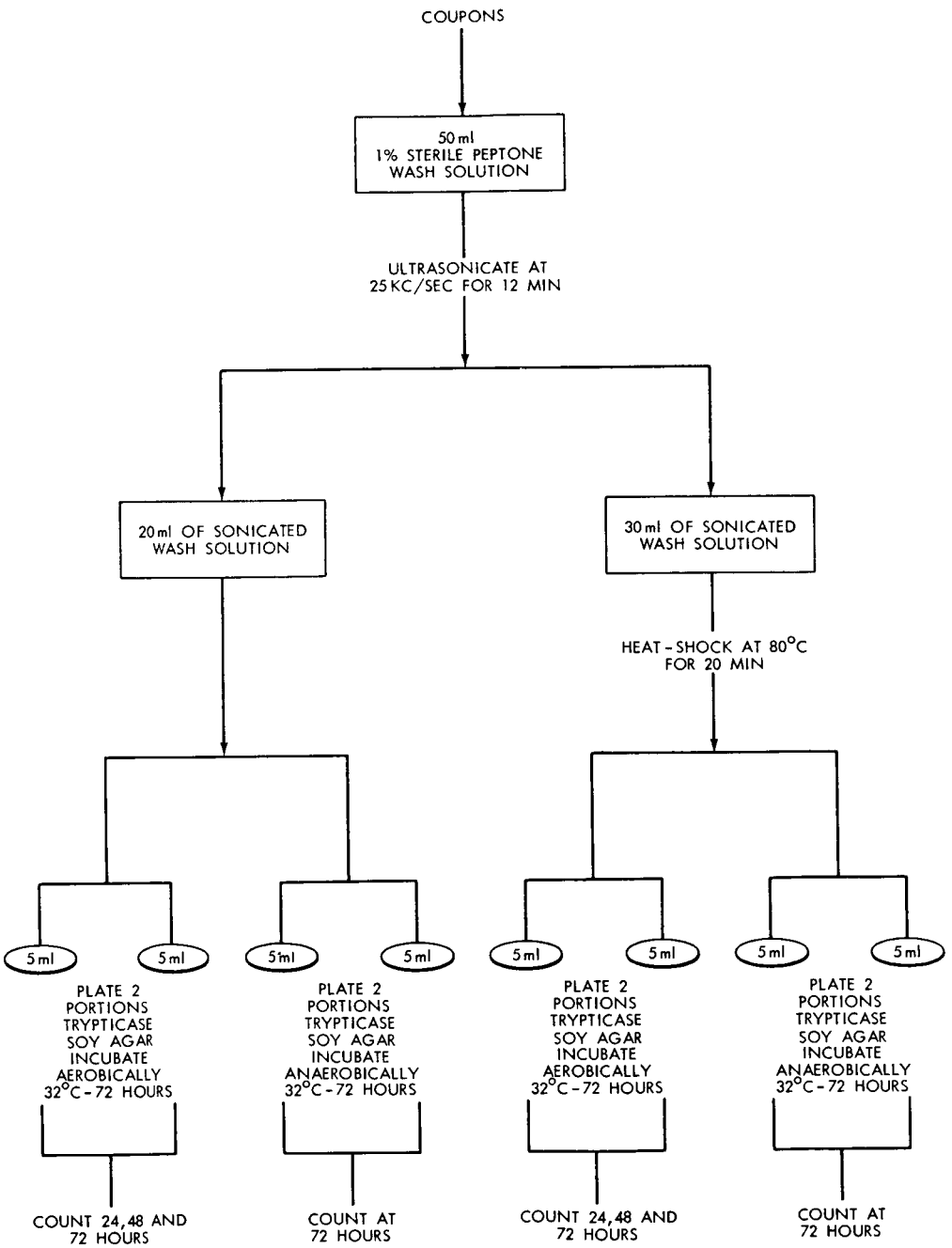



Figure 5a. Schematic of coupon assaying procedure

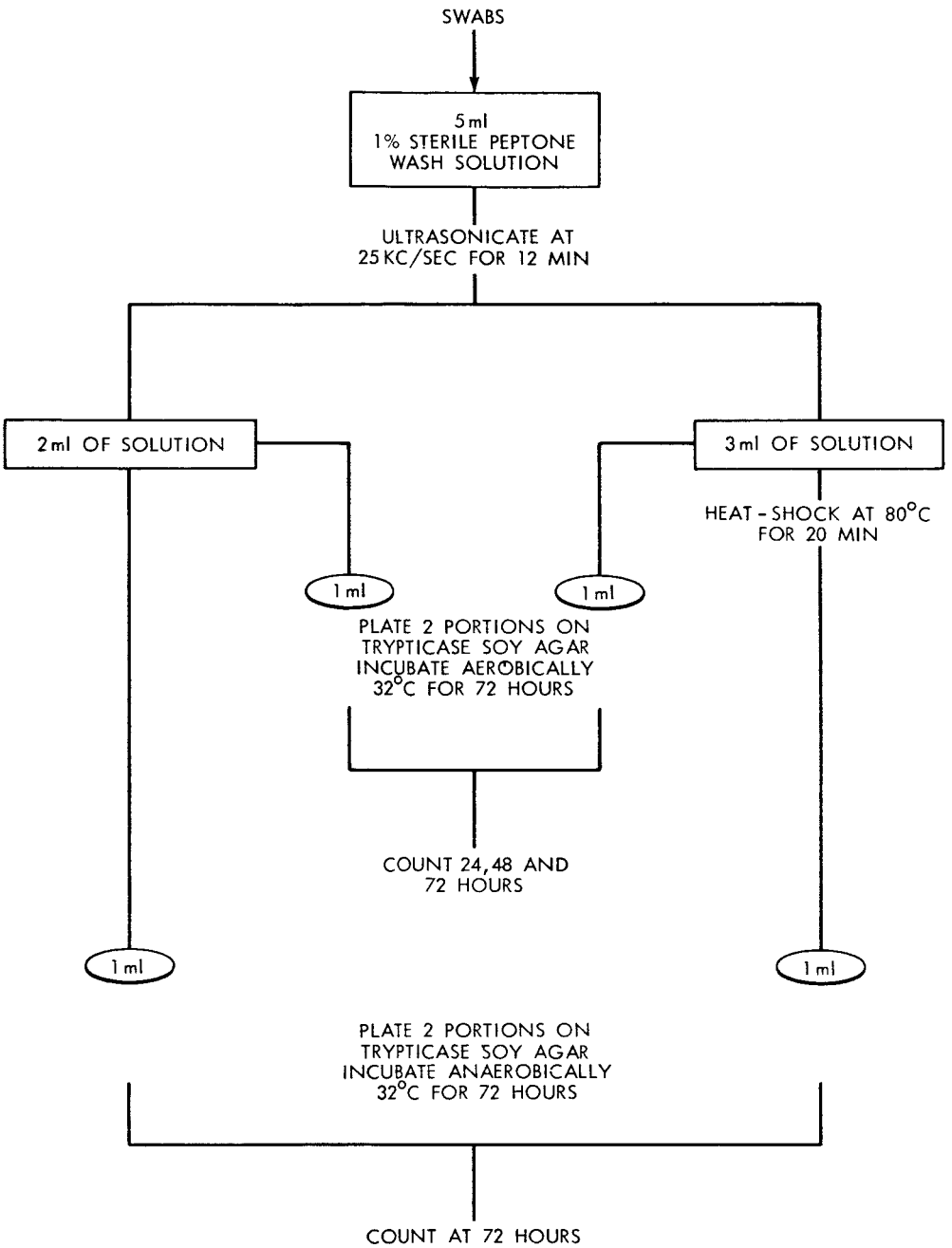



Figure 5b. Schematic of swab assaying procedure

All surfaces of the spacecraft were monitored for microbial contamination, i.e., the areas which would be occluded by an attachment, instrument or structural member and the exposed surface areas on the interior and exterior of the spacecraft. Figure 6 is a schematic that depicts the spacecraft configuration without its 4th stage retromotor, attitude control system, nutation damper and solar paddles. It also depicts the structural materials.

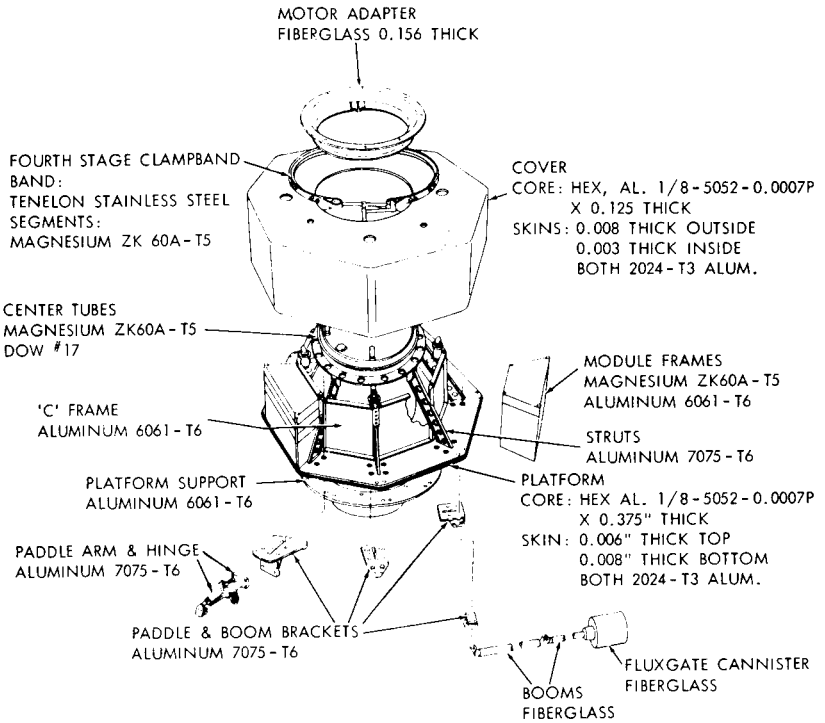



Figure 6. AIMP-E spacecraft showing spacecraft configuration and structural materials

Figure 7 depicts the manner in which the spacecraft and the various components that make up the spacecraft system were decontaminated and the manner in which the microbial samples were taken.

## Decontamination and Sampling Procedure

Electronic Circuit Modules. The electronic modules were the first component parts of the spacecraft that were decontaminated. Decontamination was achieved by first precleaning with an aerosol of ethyl alcohol to remove deposits of solder flux and/or water lacquer remaining on circuit boards after their fabrication. A control strip was then affixed to the module frame. This control strip was first sterilized in a steam autoclave before attachment. The modules were then inspected and electrical tests as required by the cognizant scientist were conducted. The modules were then delivered to the Mechanical Systems Branch at Goddard for biomonitoring and integration into the spacecraft system.

A coupon was taken from the control strip that was affixed to the module and an assay performed to determine the type and level of contamination. The module and the remaining portion of the control strip were then decontaminated by immersion in a 90% isopropyl alcohol solution. The module was agitated by hand at least three times during a 15-minute immersion. The electronic circuit module and remaining portion of control strip was then placed into a vacuum oven and subjected to a temperature of 55°C for one hour duration. A coupon was then removed aseptically and an assay performed to determine the type and level of contamination remaining on the module after decontamination process. A bacteriostatic conformal coating was then applied to the electronic

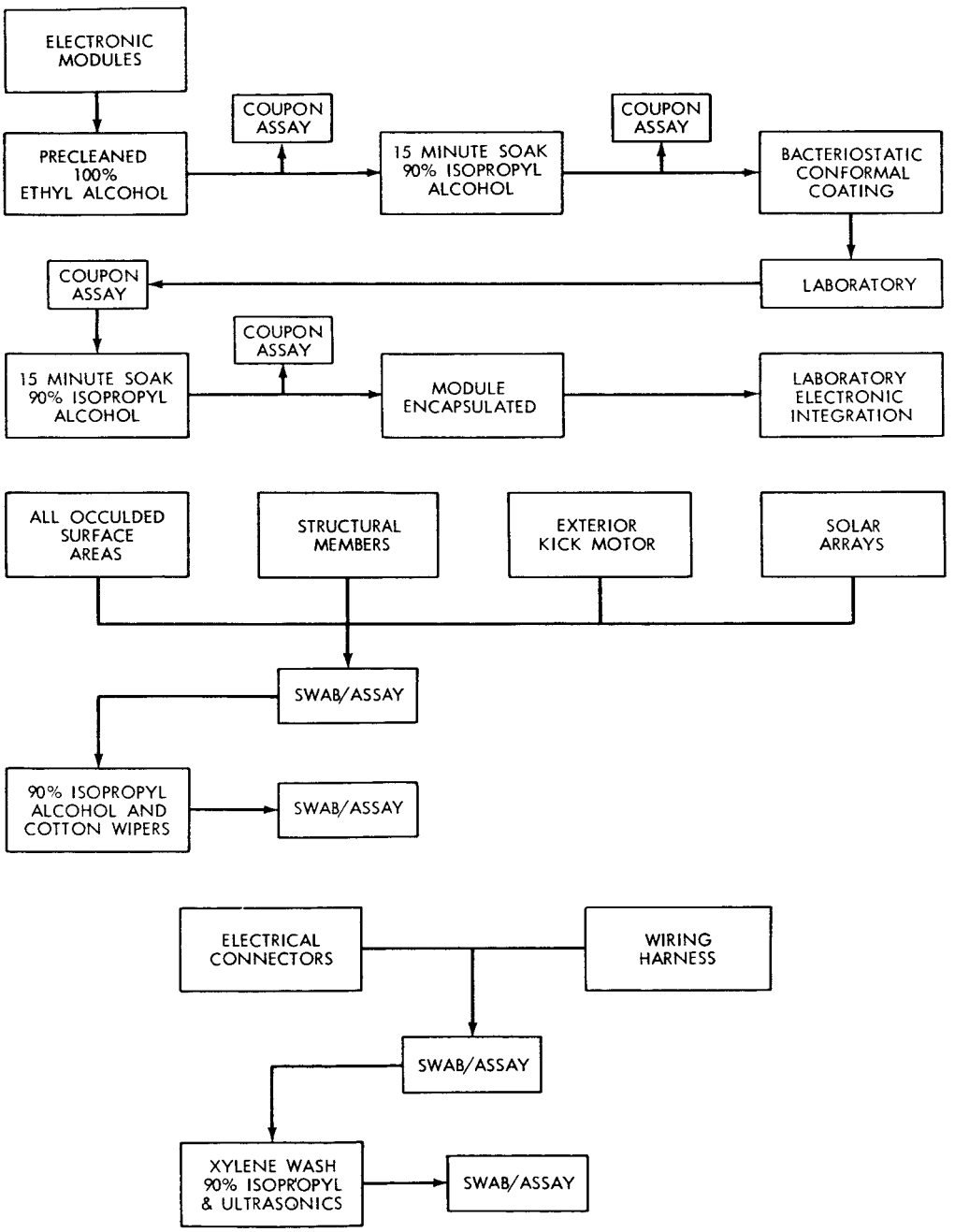



Figure 7a. Areas decontaminated, manner of decontamination and sterilization, and the method of recovering viable micro-organisms

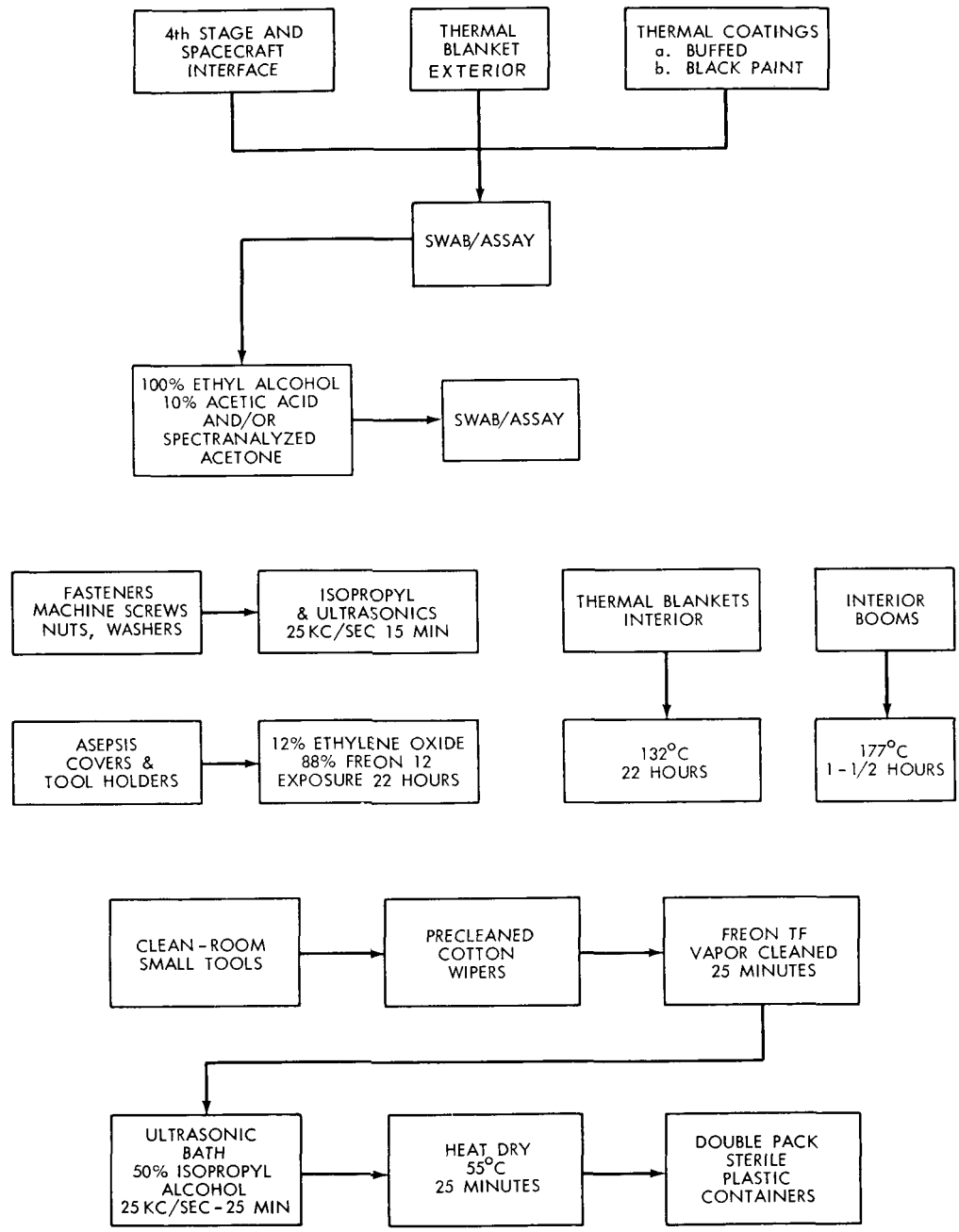



Figure 7b. Areas decontaminated, manner of decontamination and sterilization, and the method of recovering viable micro-organisms

circuits and module. Board coating had bacteriostatic qualities on the following organisms: Bacillus subtilis var niger, Staphylococcus aureus, Pseudomonas alcaligines and Corynebacterium SSP.

The circuit modules were then packaged into a protective container and delivered to the laboratory for electronic checks and testing. Upon completion of the testing the circuit modules were sent to the Mechanical Systems Branch and the above process was repeated with the exception of the conformal coating. At this time the circuit modules were encapsulated after decontamination and assaying.

Occluded Surfaces; Structural Members and Motor Exterior. Prior to occlusion by a component, a structural member or attachments such as the retromotor, the area that was to be occluded was first assayed to determine contamination level. Sterile lint-free cotton wipers and isopropyl alcohol was used to effect decontamination.

The areas to be decontaminated were first precleaned by vacuuming the area to remove any gross particles. The cotton wipers were then immersed into a 90% solution of isopropyl alcohol, and wrung damp dry by hand. The pertinent areas were then thoroughly wiped. After evaporation of alcohol, a swab sample was taken. The number of samples taken was dependent upon the area of item sampled. The larger the area the greater number of samples were taken.

Electrical Connectors and Wiring Harness. The electrical connectors and wiring harness had the rosen flux deposits removed from the connections by applying xylene with a natural bristle brush for approximately fifteen seconds. Connectors were then rinsed off in an isopropyl bath. A swab sample was taken in several areas of the wiring harness for assays to determine the contamination level. The entire wiring harness including connections was then immersed in a 90% isopropyl bath and sonicated at 40 kc/sec for approximately ten minutes. Harness was then allowed to air dry under a fume hood and swab samples taken for determining levels of contamination after decontamination.

Interface, Thermal Blankets and Coatings. The fourth stage interface, thermal blanket exterior surface and the black painted and buffed thermal coatings were decontaminated in a like manner. Prior to any decontamination a swab sample was taken for assaying the contaminated level just prior to launch. After sample was taken any stubborn stain remaining on the coatings was removed with a 10% acetic acid solution and/or swabbed with a 100% ethyl alcohol. The entire surface area was then swabbed with sterile cotton swabs that had just been immersed in acetone. Only the purest grade of spectanalyzed acetone was used. A swab sample was then taken for assay of the decontaminated surfaces. After samples were taken that particular area was again swabbed with acetone.

Thermal Blankets and Interior of Booms. The thermal blankets were first assayed with swabs to determine the contaminated level. Samples were taken from several of the layers that made up the blanket. The blankets were then sterilized with dry heat, 140°C for 16 hours. After installation on spacecraft the exposed exterior surfaces were again monitored to determine the contamination level. The blankets were sealed; therefore it was accepted that sterility of the inner surfaces was not violated. The interior of the booms were considered sterile as they were subjected to 177°C dry heat for a period of 1 and 1/2 hours during lacquer bake-on. However, the exterior of booms and thermal

blankets were exposed after sterilization and were subject to possible contamination, therefore they were again sampled, decontaminated and sampled to determine contamination levels just prior to launching spacecraft.

Miscellaneous Small Metallic Parts, Nuts, Bolts, Screws and Washers. The small miscellaneous metallic components were monitored for contamination by immersing 25% of their total number in a wash solution and plating out aliquots of solution as per the previously described technique. Decontamination of subject items was accomplished by immersion in 90% isopropyl alcohol for 15 minutes. The items were then placed into a 1% peptone wash solution and sonicated for 12 minutes at 25 kc/seconds. Samples of the solution were then plated out.

Asepsis Covers and Tool Holders. The asepsis covers for the retromotor, booms and entire spacecraft were wrapped individually in Kraft paper and inserted in a plastic container. They were then subjected to an environment of 12% ethylene oxide and 88% Freon 12 at ambient temperature of 72°F. The items were exposed at a slight positive pressure for a period of 22 hours.

Clean Room Small Tools. Tools used in the clean room during spacecraft assembly and/or field testing of the system were first precleaned by wiping off gross contamination with cotton wipers. Tools were then placed into a wire mesh basket and exposed to Freon TF* vapor cleaning for 25 minutes. They were then placed in an ultrasonic bath containing a 50% solution of isopropyl alcohol ($C_3H_7OH$) and sonicated for 25 minutes at 25 kc/sec. After removal from the solution they were placed in an oven which was preheated to 55°C. They remained in this environment for 25 minutes. All tools were then packaged and sealed in sterile plastic sheet material. These packs were again packaged so as to have tools double packed and sealed. The outer package was removed just prior to injecting tools into the Goddard Down Flow Unit for use in spacecraft assembly. To prove or disprove the adequacy of the above decontamination procedures tests were conducted on metal plates 4 x 4 square inches. These squares were left unattended and handled by several individuals so as to contaminate them as would be done in conducting an operation with tools manufactured from like materials. Dirty samples were first taken and assayed so as to determine the type and levels of contamination prior to decontamination. Swab samples were also taken after Freon Vapor Cleaning (FVC) and after the exposure to the 55°C heat drying. The test samples were placed in plastic containers and allowed to remain therein for 72 hours prior to final biosampling. The 72 hour period was selected to simulate the normal incubation period. Test samples were handled aseptically during all operations. Assays under aerobic conditions were performed to determine the reduction of micro-organisms that were achieved. The entire process was repeated with assays performed under anaerobic conditions. Table 1 depicts the results of these tests. It is to be noted that zero counts were obtained after each step in the decontamination process.

Assembly Environment (Figure 8). It is felt that the greatest source of contamination to a spacecraft system will be from the technicians themselves and from the generation of debris that occurs during the spacecraft build-up, mechanical integration and/or final assembly, and checkout of flight configuration in the field. It was therefore determined that adequate clean-room facilities should be procured that

*TRI-CHLORO-MONO-FLUORO-METHANE

Table 1

| Condition | Contaminated | | | | Decontaminated | | | |
|---|---|---|---|---|---|---|---|---|
| | Aerobic | | Anaerobic | | Aerobic | | Anaerobic | |
| | Veg | Spores | Veg | Spores | Veg | Spores | Veg | Spores |
| 1. Steel | 4 | 0 | 4 | 0 | 0 | 0 | 0 | 0 |
| 2. Brass | 139 | 0 | 27 | 0 | 0 | 0 | 0 | 0 |
| 3. Aluminum | 2 | 0 | 1.0 | 0 | 0 | 0 | 0 | 0 |
| 4. Copper | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

would allow under aseptic conditions decontamination of spacecraft and its components, biosampling of areas for assays, mechanical integration, final assembly and tests.

Spacecraft Preparation Area. This was a controlled area where the debris generating operations were performed on a component or the spacecraft structure. When it was required to custom fit a component to the structure the operations of filing, drilling or scraping of metal were necessary a shield was built to protect items not worked upon from falling metallic particles. In addition a vacuum cleaner was employed to gather loose chips as generated. The inlet of vacuum cleaner was placed in the immediate area worked upon. The spacecraft or component was again vacuum cleaned before leaving this area.

Hi-Bay Clean Room Complex (Figure 9). The Hi-bay clean-room complex consisted of a 100,000 class conventional clean-room of approximately 70 feet square, 24 feet high. Within this clean-room are class 100 portable Vertical Laminar Flow Units which are expandable in multiples of 4 x 8 units and Class 100 horizontal flow benches. The vertical flow units were used to house the spacecraft when not worked upon in the class 100,000 area. The spacecraft was precleaned each time before placing it under the down flow units. Cleaning consisted of wiping and vacuuming the surfaces. The down flow units were also used to perform instrument integration, decontamination and biosampling of components. The flow benches were used when assembling delicate mechanisms and the cleaning thereof at each stage of assembly. After the spacecraft was mechanically integrated it left the clean room area for electronic integration and/or systems environmental tests. The spacecraft was protected at that time by a strippable coating which was applied only to the exterior exposed surfaces.

Bio-Clean Room (Figure 10). Room D is a bio-clean room where satellites are decontaminated. An unusual feature of this room is a monitoring camera which photographs the satellite and personnel every 5 seconds. This feature was included to check on faulty operations that may occur while in the bio-clean room. Horizontal, laminar-flow air emanates from a 14-foot wall via modules with Cambridge high efficiency particulate air (HEPA) filter units. Filtration tests confirmed a rating for this room between 0 and 66 particles of 0.5 micron or less per cubic foot of air. Walls and ceiling are of prefabricated panels with a 4 inch insulation of plastic foam. Epoxy-coated steel forms the interior surfaces. A completely lighted ceiling gives a shadowless, 200-foot candle illumination at working levels. There are a minimum of 20 air changes per hour at a temperature range of 67°F to 77°F and

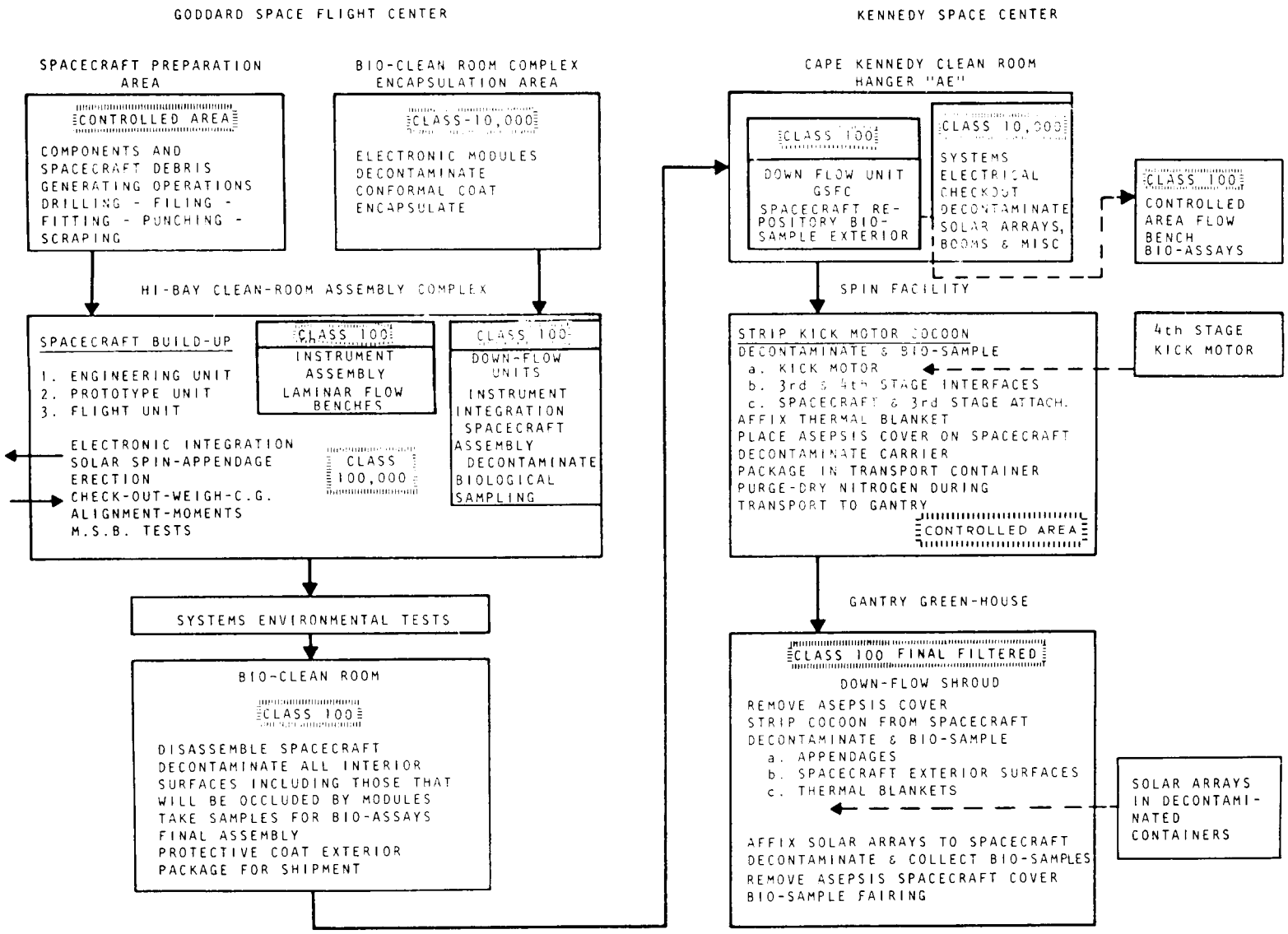



Figure 8. Assembly environment

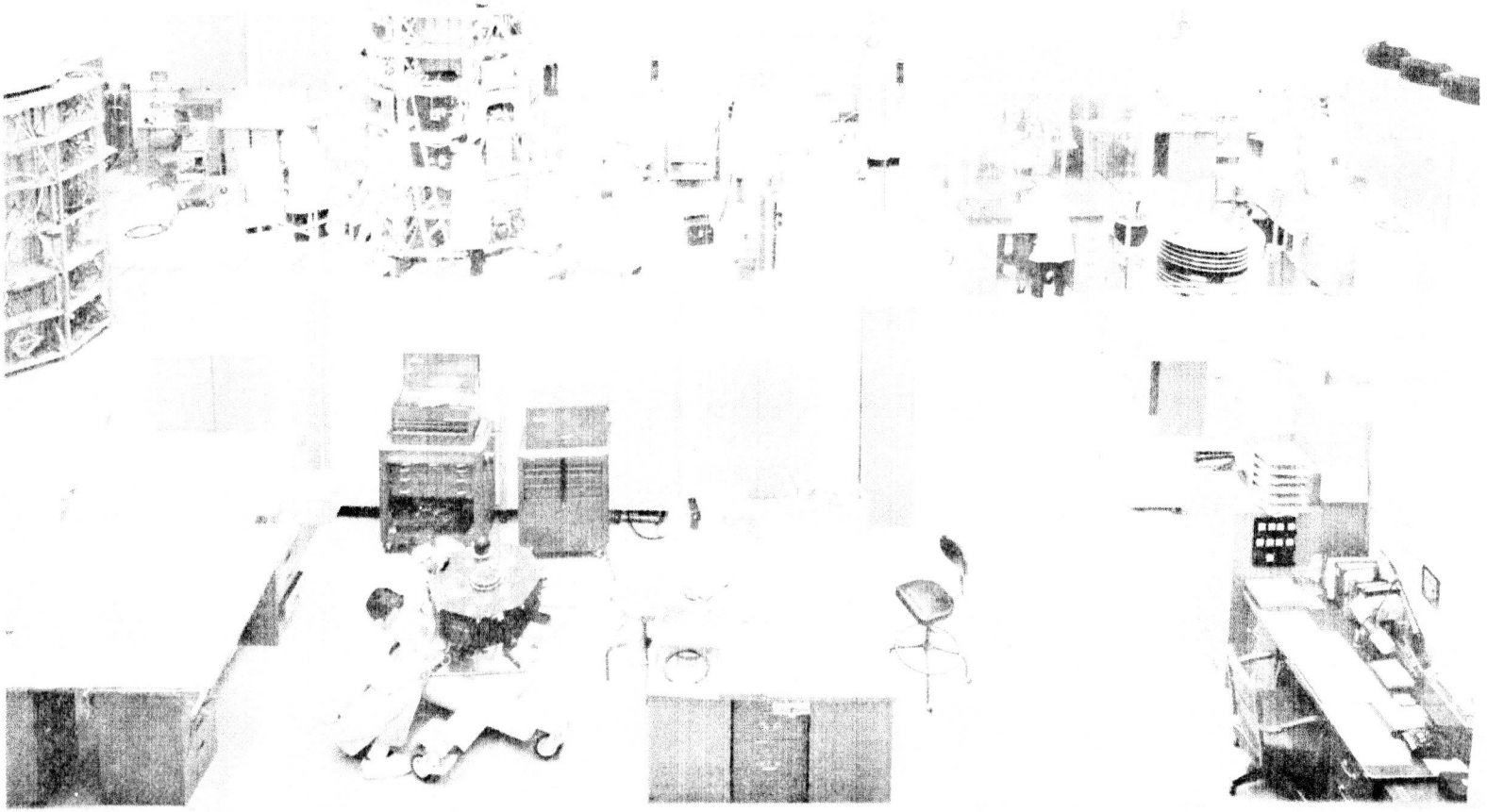


Figure 9. Hi-bay clean room complex

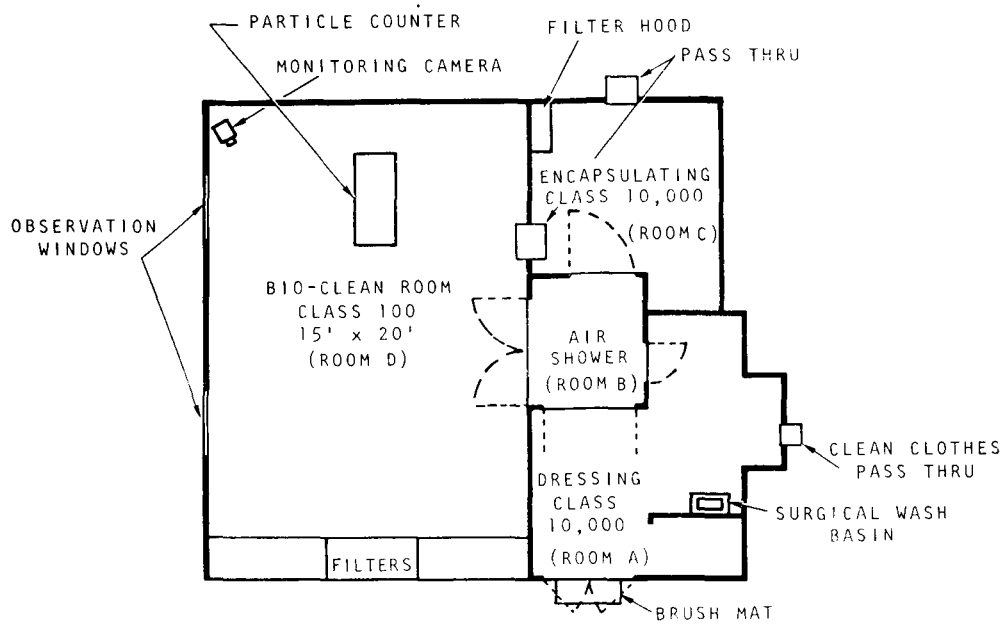



Figure 10. Bio-clean room

a relative humidity level of 40 to 45 precent. A constant temperature of 72°F was maintained. A central built-in wall-type vacuum system is provided in all four rooms, along with observation windows that are double-paned and sealed. Also included are pass-through chambers containing interlock doors to assure maintenance of a positive air-pressure when parts are brought into the room.

Upon returning from the systems environmental tests the spacecraft was returned to the Mechanical Systems Branch for disassembly, decontamination, biosampling and reassembly. These tasks were conducted in the confines of the bio-clean room complex.

The spacecraft was first precleaned with a vacuum and then wiped down with lint-free cotton wipers that had been immersed in isopropyl alcohol and wrung damp dry. The top cover was taken off and all the flight electronic modules and some instruments removed. The electronic circuit modules and instruments were then transferred to the Class 10,000 area of the clean room complex. Swab samples were taken and assays performed for determination of contamination type and levels. These items were then decontaminated with isopropyl alcohol and cotton wipers. The decontaminated circuit modules and instruments were then passed aseptically into the Class 100 area of the complex. The microbiologist then took swab samples of each item decontaminated.

The spacecraft (Figure 11) was placed upon a dolly that had previously been decontaminated and then moved into the Class 100 area of the complex. The spacecraft on its dolly remained approximately one foot from the face of the air inlet filter tank during final assembly. Personnel remained downstream of the spacecraft at all times.

The area that modules and instruments occluded was then biosampled, decontaminated and biosampled. Sampling was accomplished by swab method and decontamination accomplished with isopropyl alcohol dampened cotton wipers. The modules were then integrated into the spacecraft structure. After the integration was completed all the exposed surface areas on the

Figure 11. The spacecraft during final assembly

interior of the spacecraft were biosampled for contamination type and level, decontaminated with isopropyl alcohol and then biosampled and assays performed to determine the number of organisms remaining after decontamination. The inside of the cover was treated in a like manner and then installed on the spacecraft. Biosampling and decontamination were achieved in the same manner as the exposed interior areas. The exterior of the top cover was then cleaned and decontaminated, however, biosamples were not taken at this time. The exposed surface areas of the spacecraft were then coated with a strippable coating. The spacecraft was then placed into a container that had previously been decontaminated with isopropyl alcohol and cotton wipers. Spacecraft container was then flushed with $GN_2$ and pressurized to 15 psia with dry nitrogen. The spacecraft was then shipped by aircraft to the Kennedy Space Center for field tests and launch.

## Asepsis Control During Conductance of Field Tests (Cape Kennedy)

Sun-Spin Facility. After completion of preliminary operational checkout of spacecraft the body of the spacecraft was covered with a clean asepsis covering. The spacecraft was placed into its special container, transported to the sun-spin facility and then mounted upon the spin table. The solar paddles were then affixed to their supports. The asepsis covering remained on the spacecraft during all tests conducted in this area.

After completion of the sun-spin tests the solar paddles were removed. Swab samples were then taken on the solar paddles for assays to determine the biocontamination type and levels in the contaminated state.

The spacecraft was packaged and then transported to the air-lock of the AE clean room.

Cape Kennedy Clean Room Facility (Figure 12). The Cape Kennedy Class 10,000 clean room, located in the AE building, was used to house the Goddard Class 100 laminar downflow unit.

The laminar downflow unit and the exterior of the spacecraft container were first decontaminated before moving them into the clean room.

The Goddard Downflow Unit which housed the AIMP-D spacecraft during various tests and experiments was decontaminated and assembled in the airlock, Hi-bay clean room (Figure 9). The procedures employed include the following: decontaminating the spacecraft dolly which also involved removing the strip coating; preparation of ground support equipment (GSE) which entailed removing connecting cables, air inlet filters, all tape and paper units, and filling the voids in instrument racks with cover plates, and replacing air inlet filters with new filter material; and decontaminating electronic equipment connection cables by passing them between two sponges that were soaked in an alcohol solution. Mitocs, telephones, hand tools, and lead pigs that contained radioactive sources were treated in the same manner.

The spacecraft was removed from its container and placed upon the previously decontaminated dolly. The dolly and spacecraft were then placed under the downflow unit and remained there during the field checkout tests.

Authorized persons who were conducting tests and experiments or working in any of the clean room areas were subjected to the following partial list of regulations:

- Personnel with respiratory malfunctions, skin ailments, colds, and severe sunburn were not permitted in clean room areas.
- Test fixtures, tools, jigs and assembly fixtures that were necessary to perform specific tasks were permitted.
- No abrasives, e.g., file, crocus cloth, etc., and no shredding or masking tapes.
- Exposed parts or components were never left on work benches.

- Approved clean room garments were worn in various clean room areas as previously mentioned.
- Smoking or eating was not permitted.
- Scratching of the head, eyebrows, and other exposed areas of the skin was forbidden.
- Only 5 persons were allowed in the Hi-bay clean room or any of the other clean room areas at any one time.
- No more than two persons were allowed under the GSFC Downflow Unit at the same time.
- Hand tools that were not in use were stored in a decontaminating solution.

The clean room regulations as stated above were necessary to assure asepsis handling of the AIMP spacecraft during experiment check-out phases.

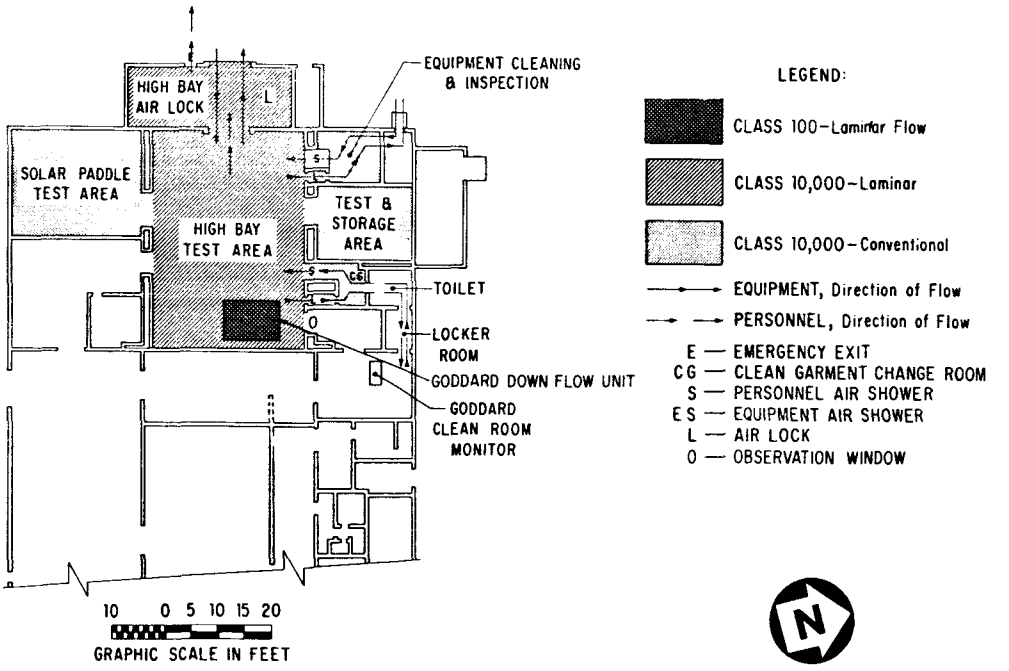



Figure 12. Spacecraft clean rooms, Building "AE"

Air Sampling. Trays of stainless steel strips (1x2 inches) were positioned on one side of the laminar crossflow, Class 10,000 clean room, upstream, downstream and midway of the clean room and one tray was placed in the Class 100 downflow unit bench high at working level. Six strips were recovered once a week from each location and assayed for aerobic and anaerobic vegetative and sporalitive viable life. Air samples were collected on two occasions; prior to introducing spacecraft into downflow unit and during field checkout testing. Renier slit samplers with a one-hour clock were used. Air was drawn into the samples

at a rate of 1 cubic foot per minute for 60 minutes. Glass plates (150x20 mm) containing 60 ml of Tripticase Soy Agar were used as collectors in the samplers. Air samples were placed approximately at bench top level (3 feet from the floor) in three different positions--upstream, downstream and in the Goddard downflow unit.

Table 2 lists the microbial contamination in the air of the clean rooms which housed the AIMP-E flight spacecraft during field checkout tests, as well as the number of personnel in the room during sampling period.

Table 2

Microbial Contamination in the Air of Laminar Flow Clean Rooms Over a 7 Hour Period

| Hour | Viable Particles Per Cubic Foot Per Hour | | | | | |
|---|---|---|---|---|---|---|
| | Crossflow Room Downstream | | Crossflow Room Center | | Crossflow Room Center | |
| | Date: 6/9/67 | | Date: 6/9/67 | | Date: 6/9/67 | |
| | Count Part./ft$^3$ | Personnel | Count Part./ft$^3$ | Personnel | Count Part./ft$^3$ | Personnel |
| 1 | 0 | 0 | 0 | 0 | 0 | 0-1 |
| 2 | 0 | 0-2 | 0.033 | 0-2 | 0.050 | 0-2 |
| 3 | 0 | 0-1 | 0 | 0-1 | 0.033 | 0-1 |
| 4 | 0 | 0-2 | 0.016 | 0-2 | 0 | 0-1 |
| 5 | 0 | 0-1 | 0 | 0-1 | 0 | 0-3 |
| 6 | 0.016 | 0-1 | 0 | 0-1 | 0 | 0-2 |
| 7 | 0.083 | 0-1 | 0 | 0-1 | 0 | 0-2 |
| Average Viable Particle/ft$^3$ | 0.0141 | | 0.007 | | 0.0119 | |

Microbial Contamination in the Air of Laminar Flow Clean Rooms Over a 4 Hour Period

| Hour | Viable Particles Per Cubit Foot Per Hour | | | | | |
|---|---|---|---|---|---|---|
| | Downflow Downstream | | Downflow Upstream | | Crossflow | |
| | Date: 6/27/67 | | Date: 6/27/67 | | Date: 6/27/67 | |
| | Count Part./ft$^3$ | Personnel | Count Part./ft$^3$ | Personnel | Count Part./ft$^3$ | Personnel |
| 1 | 0.166 | 0-2 | —* | 0-2 | 0.050 | 3-5 |
| 2 | 0.033 | 1 | 0.033 | 1 | 0.050 | 0-3 |
| 3 | 0 | 0-3 | 0.033 | 0-3 | 0.016 | 0-5 |
| 4 | 0.016 | 0-2 | 0 | 0-2 | 0.016 | 1-4 |
| Average Viable Particle/ft$^3$ | 0.0537 | | 0.0220 | | 0.0330 | |

*Not Countable

Table 3 lists the microbial fallout on the stainless steel strips over a 4-week period.

Table 3
Microbial Fallout on Stainless Steel Strips

| Week of Exposure | Viable Organisms Per Square Foot | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Downflow Room | | | | Crossflow Room | | | | | | | | | | | |
| | | | | | Upstream | | | | Center | | | | Downstream | | | |
| | Vegetative Organisms | | Spores | | Vegetative Organisms | | Spores | | Vegetative Organisms | | Spores | | Vegetative Organisms | | Spores | |
| | Aerobic | Anaerobic | Aerobic | Anaerobic | Aerobic | Anaerobic | Aerobic | Anaerobic | Aerobic | Anaerobic | Aerobic | Anaerobic | Aerobic | Anaerobic | Aerobic | Anaerobic |
| 1 | 0 | 0 | 0 | 0 | 4 | 6 | 0 | 0 | 3 | 0 | 0 | 0 | 25 | 2 | 0 | 0 |
| 2 | 1 | 1 | 2 | 3 | 5 | 4 | 7 | 1 | 6 | 6 | 4 | 2 | 2 | 2 | 3 | 0 |
| 3 | 3 | 0 | 0 | 0 | 8 | 0 | 3 | 0 | 0 | 0 | 8 | 0 | 3 | 0 | 2 | 0 |
| 4 | 0 | 1 | 0 | 0 | - | - | - | - | - | - | - | - | - | - | - | - |

After completion of the electronic and instrument checkouts the spacecraft was removed from the Goddard laminar downflow unit and placed into its container while in the AE Class 10,000 clean room. The container had previously been decontaminated with alcohol and cotton wipers.

Spin Balance Facility. The spacecraft fourth stage and retromotor interfaces were swabbed sampled for assays to determine contaminated state. They were then decontaminated and swabbed sampled for assays to determine the bioburden remaining after decontamination process. The thermal blanket that had previously been sterilized was then placed over the fourth stage retromotor. The third stage and spacecraft interfaces were treated in a like manner as the fourth stage interfaces. The solar paddles were then attached to the body of the spacecraft. The third and fourth stage vehicles and the spacecraft in its flight configuration were dynamically and statically balanced as a unit. After the balance tests the paddles were removed and transported back to the AE clean room for final decontamination and bioassays.

An asepsis cover, previously sterilized with ethylene oxide compound, was placed over the entire spacecraft. The entire unit was then placed in a previously decontaminated transfer container. The container was sealed and was then pressurized with a slight positive pressure of gaseous dry nitrogen. The canned unit was then transported to the gantry and affixed to the second stage vehicle.

Gantry Operations. The transfer container was removed and the asepsis bag was allowed to remain intact over the spacecraft until the

air cooling hat-shroud was placed in operation. The cooling hat shroud was first LOX compatible cleaned and decontaminated with isopropyl alcohol before assembly on the gantry. Verification that the filtered air supplied to the cooling hat shroud was Class 100 was authenticated by chemical engineers from PanAm using a Royco particle counter. Three tests were run. One test for one minute and two runs of 10 minutes each. The filtered air entered from the top of the cooling shroud. It was temperature controlled and passed through a diffuser designed to assimilate a vertical laminar flow of air over the spacecraft. The sides of the cooling hat shroud were rolled up to permit mating of the nose cone fairing which was fitted to the Delta vehicle. During this operation the asepsis cover remained on the spacecraft to protect it from fall-out of particulate matter. During this operation the flow of air was continued. The sides of the shroud were let down after the fitting.

The nose cone fairing was decontaminated and packaged prior to delivery to gantry and/or assembly on vehicle.

The solar paddles were decontaminated in the Goddard downflow unit, placed into containers that were previously decontaminated with isopropyl alcohol. Prior to launch the asepsis covers were removed and all the protective strippable coatings were removed. All exposed surfaces were then swab sampled for bioassays in contaminated state.

The thermal coatings were decontaminated with triple-distilled acetone, spectranalyzed grade. However, all stubborn deposits were first removed with a 10% acetic acid solution and 100% ethyl alcohol. All other surfaces were decontaminated with a 90% solution of isopropyl alcohol. Prior to installation of the solar paddles, all mating surfaces were decontaminated with isopropyl alcohol prior to occlusion. Tie-down cords were then affixed.

All representative areas were biologically sampled so that assays could be performed to determine burden at time of launch.

The asepsis cover was replaced on the spacecraft. Nose cone fairing asepsis covering was then removed.

One-half of the nose cone fairing that was previously decontaminated was mated to the vehicle. The filtered conditioned air umbilical was connected to this section of nose cone fairing. The spacecraft asepsis cover was removed just prior to mating the other half of the nose cone fairing.

The spacecraft was constantly bathed with Class 100 filtered and temperature conditioned air until separation of umbilical at time of lift-off.

## Results

On the basis of the Goddard Bio-Records (see Table 4), it was determined that the surfaces of the AIMP-E spacecraft contained not in excess of $9 \times 10^5$ micro-organisms prior to decontamination, and not in excess of $2.7 \times 10^4$ micro-organisms after the decontamination process. This constituted a 97% reduction of organisms. The estimation of viable internal burden of components (see Table 5) was based upon past history and known manufacturing environments. It was determined that, of the total viable life remaining in the components, (see Table 7) 10% would

Table 4

| Area Class | Total Area ($in^2$) | Compilation of Counts of Viable Organisms on Surfaces | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Contaminated | | | | Decontaminated | | | |
| | | Aerobic | | Anaerobic | | Aerobic | | Anaerobic | |
| | | Veg. | Spores | Veg. | Spores | Veg. | Spores | Veg. | Spores |
| Occluded "A": Electronic Modules | 8759 | 212899 | 62232 | 26858 | 25547 | 5937 | 3251 | 4099 | 55 |
| Occluded "B": Surfaces that Module Frames Occlude | 1138 | 4033 | 0 | 5914 | 0 | 72 | 0 | 3450 | 0 |
| Occluded "C": Exterior of Module Frames | 5813 | 56300 | 4489 | 9923 | 85 | 20 | 2030 | 192 | 0 |
| Occluded "C": Other Interior Exposed Surfaces of the Spacecraft that the Cover Occludes | 5195 | 20001 | 17467 | 898 | 40206 | 79 | 178 | 0 | 369 |
| Interior Surfaces "D": Other Interior Surfaces of the Spacecraft —1 Body —2 Motor —3 Assembly Occluded | 8490 | 63174 | 6517 | 8747 | 1750 | 1985 | 502 | 38 | 12 |
| Exterior Surfaces "E": Exterior Surfaces of the Spacecraft —1 Body —2 Motor —3 Assembly Occluded | 71437 | 228329 | 80818 | 19321 | 4344 | 3671 | 420 | 281 | 545 |
| Final Totals of Contamination of the AIMP-E Spacecraft | Total 700.2 sq.ft. | 584736 | 171523 | 71661 | 71932 | 11764 | 6381 | 8060 | 981 |
| GRAND TOTALS | | $9 \times 10^5$ | | | | $2.7 \times 10^4$ | | | |

Table 5

Compilation of Viable Organisms Contained Within Components

| Components | Estimated Range | Number of Components | Accumulative Total x $10^3$ | |
|---|---|---|---|---|
| | | | Low | High |
| Resistors | 0-1 | 11612 | 0 | 11.6 |
| Capacitors | 10-100 | 3153 | 31.5 | 326.9 |
| Diodes | 0-1 | 4005 | 31.5 | 330.9 |
| Transistors | 0-1 | 3164 | 31.5 | 334.1 |
| Relays | 100-1000 | 15 | 33.0 | 349.1 |
| Crystals | 0-1 | 1 | 33.0 | 349.1 |
| Inductors | 0 < 100 | 148 | 33.0 | 363.0 |
| Toroids Transformers | 0 < 100 | 117 | 33.0 | 375.6 |
| Batteries | 0 | 0 | 33.0 | 375.6 |
| Metals | 0 | 0 | 33.0 | 375.6 |
| Tubes | 0 | 4 | 33.0 | 375.6 |
| Explosives | 10 | 8 | 33.1 | 375.7 |
| Foam | 1/ml | 14727 ml | 47.8 | 390.4 |
| Nylon-Dacron | 0 | 876 | | |
| Teflon Insulation | 0 | 16 | | |
| Magnetic Cores | 0 | 0 | | |
| MOSFETS | 0 | 747 | | |
| Pots | ? | 17 | | |
| Flat Paks | 0 | 551 | | |
| Fuses | 0 | 15 | | |
| Thermistors | 0 | 35 | | |
| Estimated Total - Internal Burden | | | 47.8 | 390.4 |
| Average Internal Burden | | | 219.0 | |

be spore forms. Of this 10% approximately two-thirds would be aerobic and the remainder anaerobic.

As a result of the overall evaluation (see Table 6), it was determined that at time of launch the AIMP-E spacecraft contained not in excess of $2.5 \times 10^5$ organisms. Of these an estimated $2.2 \times 10^5$ organisms were contained inside the components and foam encapsulant, and $2.7 \times 10^4$ organisms on the surfaces, $7.4 \times 10^3$ of these were spores.

The AIMP-E spacecraft achieved a successful orbit with a life expectancy of three years and will have 1440 cycles temperature change between -45°C and +50°C in an ultra-high vacuum. Under this environment the spore population on the exposed surfaces of the spacecraft should be reduced to $1.89 \times 10^{-9}$ at time of lunar impact, and all vegetative life assumed no longer to exist, only the components internal spore burden ($2.2 \times 10^4$) would remain.

The Planetary Quarantine Officer, NASA Headquarters, recommended certification of the AIMP-E spacecraft based upon the evaluation of records maintained at the Goddard Space Flight Center, visual observations of control procedures, and assessment of the microbial environment of the spacecraft while in residence at the Eastern Test Range.

Table 6

| Microbial Load at Launch<br>AIMP-E Spacecraft | |
|---|---|
| Type Load | Contamination Level |
| Internal Burden | $2.2 \times 10^5$ |
| Surfaces | $2.7 \times 10^4$ |
| Total Load | $2.5 \times 10^5$ |

Table 7

| Estimated Spore Loading at Launch & Lunar Impact | | | |
|---|---|---|---|
| Area | Aerobic | Anaerobic | Totals |
| Surfaces | $1.3 \times 10^4$ | $1.9 \times 10^3$ | $1.5 \times 10^4$ |
| Internal Burden | $1.5 \times 10^4$ | $7.3 \times 10^3$ | $2.2 \times 10^4$ |
| Grand Totals | $2.8 \times 10^4$ | $9.2 \times 10^3$ | $3.7 \times 10^4$ |
| Remaining at Lunar Impact | $1.89 \times 10^{-9}$ | | $2.2 \times 10^4$ |

# 2. ASSAY TECHNIQUES FOR PLANETARY QUARANTINE

by

M. S. FAVERO
U. S. Public Health Service
Phoenix, Arizona

We will give a brief rundown on the research activities at the National Communicable Disease Center of the Public Health Service, Phoenix, and also describe some of the activities at our assay laboratory at Cape Kennedy. There is a standard method which is described in a NASA publication entitled Standard Methods for the Microbiological Examination of Space Hardware, and this was undoubtedly the protocol that Mr. LeDoux was using.

We are usually talking about two programs. One is the Lunar program, for which a sterility requirement is not present. However, there is a clean requirement for a maximum number of organisms above which the contamination level cannot exceed. The other is the Voyager program, for which the space hardware must be sterile. In the Voyager program, we must certify sterility without actually doing a sterility test in contrast to other fields, such as the pharmaceutical industry and hospitals, where, when one wants to prove sterility, one can test a certain number of items of a particular product. We cannot test because our tests are to destruction, and we cannot test the final spacecraft. So the primary rationale is to use assay techniques which will give some fairly good estimation of the microbial load on the spacecraft. Once we know this, a particular heat cycle will be used. The more organisms that are present at a given temperature, the longer the heat treatment must be.

There are two general categories of assays. One concerns the spacecraft, the other, the intramural environment or the areas in which the spacecraft is assembled. For the intramural environment, we have been using Renier slit air samplers. There have been recent questions as to the actual value of doing air sampling, since it does not tell too much about the contamination load on the spacecraft. But air sampling does give a good idea of the type of environmental control that is used.

Most of the emphasis these days is placed on direct sampling of the spacecraft, as well as using the collecting surfaces in the environment. The idea is to simulate the surface of the spacecraft to determine how many micro-organisms accumulate there. Mr. LeDoux, in his protocol, showed that we do use ultrasonic energy for two reasons. First, it efficiently removes microbial contaminants from a surface, especially under worst case conditions. The surviving micro-organisms on a surface that has been treated with alcohol or with heat will stick tenaciously to the surface, and manual shaking or mechanical agitation will not remove them. However, ultrasonic energy (25 kc/sec, 300 watts, 2.5 watts/in$^2$ of bottom tank surface area) removes these organisms. Second, ultrasonic energy tends to break up clumps of micro-organisms. In any

enumeration system in microbiology, we are always plagued with these clumps. They are like a bunch of grapes. If the bunch of grapes is not broken up, the bunch forms one colony. This is what we count--colonies of organisms. However, if one colony is broken up, it might form a thousand colonies. So, if the assay system does not discriminate and is not consistent, the subsequent results are not consistent nor very precise. Ultrasonic energy breaks up micro-organisms into hopefully their smallest colony forming unit size.

A method that is currently being evaluated and that was originally developed by Sandia Corporation, Albuquerque, and one that will be discussed by Mr. Morris is the vacuum probe. This instrument has a potential of allowing us to sample a greater area of the spacecraft. As Mr. LeDoux pointed out, we can either use detachable strips that are placed on the spacecraft or a swab, but with all of these we are only sampling a small part of the spacecraft.

Another problem that will have to be contended with in the Voyager program is the recovery of micro-organisms from solid materials. This problem is probably unique to planetary quarantine because in other fields of microbiology, we are not concerned with organisms within solid materials, such as transistors, capacitors, and plastics. The philosophy is to reduce the particle size of the solid to a size small enough to insure the release of the entrapped organisms, but to do it in such a manner that the organisms are not killed in the process. This is probably the area that needs the most amount of work because there has been very little headway in the research done in the past 3-4 years. We use a Pico-Blender mill; although its accuracy is not the best, it is relatively precise. It does kill a certain number of organisms, but it usually kills about the same number each time, depending on the material. We have developed a model system that will be described later by Mr. Peterson, who will discuss the probability of release of micro-organisms from solids during crushable impact.

Another area of research in which we have been involved for the past few years is contamination levels in clean rooms. A bioclean room is now being used as a clean room. However, 3-4 years ago, we were still talking about conventional clean rooms, about showers, and about surgical garments. After several groups throughout the United States did a series of comparative tests, we found that a Class 100 clean room is best for controlling airborne contamination. We have also compared contamination levels in hospital operating rooms by using the same techniques as those in a spacecraft assembly area. We found in hospital operating rooms the highest levels of contamination that we have ever found, higher than in factory areas, both on surfaces and within the air. Whereas a few years ago many microbiologists were saying, "Let's treat the spacecraft as a patient is in surgery," they now say, "Let's treat the patient as we do the spacecraft."

Still another area of research is determining the dry heat resistance of micro-organisms. This work is being done primarily at the Public Health Service laboratory in Cincinnati and at Michigan State University. Before NASA's participation, most of the information on heat resistance was relegated to moist heat, such as in the canning industries, and not too much kinetic information was known about dry heat. We have learned that the water activity or relative humidity is extremely important. One can have an RH of 10 percent versus 80 percent. Are both of these dry heat? It is still an open question, but we now know that the old concept of "the drier the heat, the more resistant the spores" is not true. RH in the range of 35-45% is optimum for resistance. Anything drier than that the organisms become more sensitive.

This particular research is important because it is on these results that the final sterilization cycle will be based. The cycle that is most talked about today is 125°C for 24½ hours. However, when one heats a spacecraft in an oven to that temperature, it is going to be done slowly. On the way to this temperature, organisms are also dying, and as it cools down, more organisms are dying. Consequently, the concept of total fatality will be integrated into the sterilization cycle.

The sterility control laboratory at Cape Kennedy was opened in April 1966, and our primary mission there is to do the field work of the research group in Phoenix. At present, we are concerned with the Lunar spacecraft unmanned program, although recently we have become involved with the Apollo program. We work very closely with Mr. LeDoux on the AIMP's. We have come up with these values of microbial loading on Lunar spacecraft in terms of bacterial spores and not of total micro-organisms:

Surveyor 2 - $3.3 \times 10^5$ spores

Surveyor 4 - $2 \times 10^5$ spores

Lunar Orbiter 2 - $1 \times 10^7$ spores

Lunar Orbiter 7 - $1 \times 10^7$ spores

So when one reaches the values above without any sterilization processes, but with just decontamination, that is very good.

The primary philosophy to be followed on the Voyager program is to maintain the microbial load at extremely low levels so that we do not have to heat the vehicle too long. To do this, not only does one have to decontaminate periodically with things such as isopropyl alcohol, ethylene oxide, and short heat soaks, but also one must employ a clean environment and use it constantly. One cannot go from a Class 100 clean room to a refueling area that has the same number of micro-organisms as a barnyard. If one is going to use Class 100 conditions, they must be Class 100 all the way.

# 3. THE VACUUM PROBE FOR REMOVING ORGANISMS FOR COUNTING

by

M. E. MORRIS

Sandia Corporation
Albuquerque, New Mexico

## Introduction

Space vehicles landing on planets designated as biological preserves must be sterilized. To obtain the probability of sterility exhibited in the Sherry-Trauth mathematical model without harming any part of the space vehicle's hardware, an optimum heat cycle for sterilization must be designed. The major parameter associated with the design of this cycle is the number of micro-organisms present on the hardware surfaces before terminal sterilization.

There are two approaches which may be taken to obtain the microbial burden. The first of these, which is the most difficult to perform, is to assay the surfaces directly. The second approach is to develop a physical model (a set of surfaces with defined characteristics) best suited for the collection of micro-organisms. This physical model could be situated so that the microbial contamination of the model per unit area would always exceed that of the actual spacecraft. The results from the assay of the physical model could be used to establish an upper bound on the number of microbes on the space hardware. regardless of the technique chosen, some means must be available for an accurate and reliable assay of large surface areas with small microbial loading densities.

The recovery of micro-organisms from surfaces has been studied by microbiologists since the early part of this century. During this period, four basic methods have evolved: the agar overlay method, the agar contact method, the swab-rinse method, and the rinse method. Each method has individual disadvantages in addition to a common disadvantage of not being designed for use in situations involving small numbers of microbes on large surfaces. The agar overlay method and the agar contact method (Rodac plate method) have been shown to deposit residual nutrient materials on the surface being assayed. Use of the swab-rinse method and the rinse method is limited to surfaces which are not moisture sensitive. The rinse method may also leach toxic substances, producing bacteriostatic or bacteriocidal conditions in the culture medium.

The settling strip method is available for estimating the viable contamination on surfaces. With this method sterile stainless steel strips or strips of other materials are placed in the same environment as the space hardware, and after a determined period of environmental exposure, the strips are assayed for microbial contamination. The criticisms of this method are that it is indirect and that it has poor resolution when the amount of microbial contamination is small.

Since no method was available which would accurately assay small numbers of micro-organisms on large test surfaces in ultra-clean environments, a development project was started to provide an instrument with the desired capabilities. The device developed is based on the principles of gas dynamics and has been named the vacuum probe.

## Description of Apparatus and Experimentation

An assembled view of the vacuum probe is shown in Fig. 1; a disassembled view, in Fig. 2. The unit consists of (1) a special teflon probe tip, as shown in Fig. 3, (2) a cone designed to give an even expansion of air, (3) a base cap from a Gelman 2-inch stainless steel filter holder, along with copper mesh filter-backing pad, and a circular filter retainer ring; (4) membrane filters of 0.45 micron pore size; and (5) a Nester glassware clamp attached to a handle for holding the probe.

The assembled vacuum probe (Fig. 1) is attached to a vacuum pump with a hose at the air connection on the base cap. When the probe is placed perpendicular to a surface and in contact with it (Fig. 4), an orifice is created, as shown in Part C of Fig. 3. If the flow rate is approximately 2 cfm, the air at the orifice is at a critical flow velocity. When the air is at the critical flow velocity, the probe efficiently removes particles down to 1 micron diameter.

The bacteria or other micron-sized particles which lie on a smooth surface are difficult to remove because of the relatively strong adhesive forces and because of the difficulty in disturbing the boundary layer of air near the surface. The particles lie well within the lower regions of this boundary layer. The air entering the probe tip disturbs this boundary layer of air and raises the bacteria into the moving airstream. Once caught by the moving air, the bacteria are carried on through the probe and may be treated as any other airborne particle.

At this point, a description of the procedure used to evaluate the probe will be given. The test surfaces were inoculated with an essentially reproducible number of micro-organisms. One-half of the test surface was vacuumed with the probe (Fig. 4), and the other half of the test surface was used as a control for the experiment. The test surface was overlaid with agar and allowed to cool for 60 minutes. The agar-plated test surface was covered with sterile saran wrap (Fig. 5), inverted, and incubated at 32°C for 72 hours (Fig. 6). The colonies counted allowed a percentage removal to be calculated. The filter from the vacuum probe was removed, placed on a layer of agar in a Petri dish, and overlaid with more agar. After cooling, the dish was inverted and incubated at 32°C for 72 hours (Fig. 7). This data, along with the number of colonies on both halves of the test surface, allowed a percentage assay to be calculated.

The specific test surfaces used were flat bottomed aluminum pens with dimensions up to 11" x 16" and raised edges up to 1/2" in height. In addition, 12" x 16" stainless steel plates without raised edges and 9" x 14" glass pans and 11" x 16" teflon-coated pans with raised edges were used.

Several additional experiments were performed to insure that the planned procedure was reliable. Petri dishes were placed alongside the pans while the pans were being inoculated. When the dishes were overlaid with agar, incubated, and counted, they showed that the test surfaces used caused no bacteriocidal or bacteriostatic effects. The plates

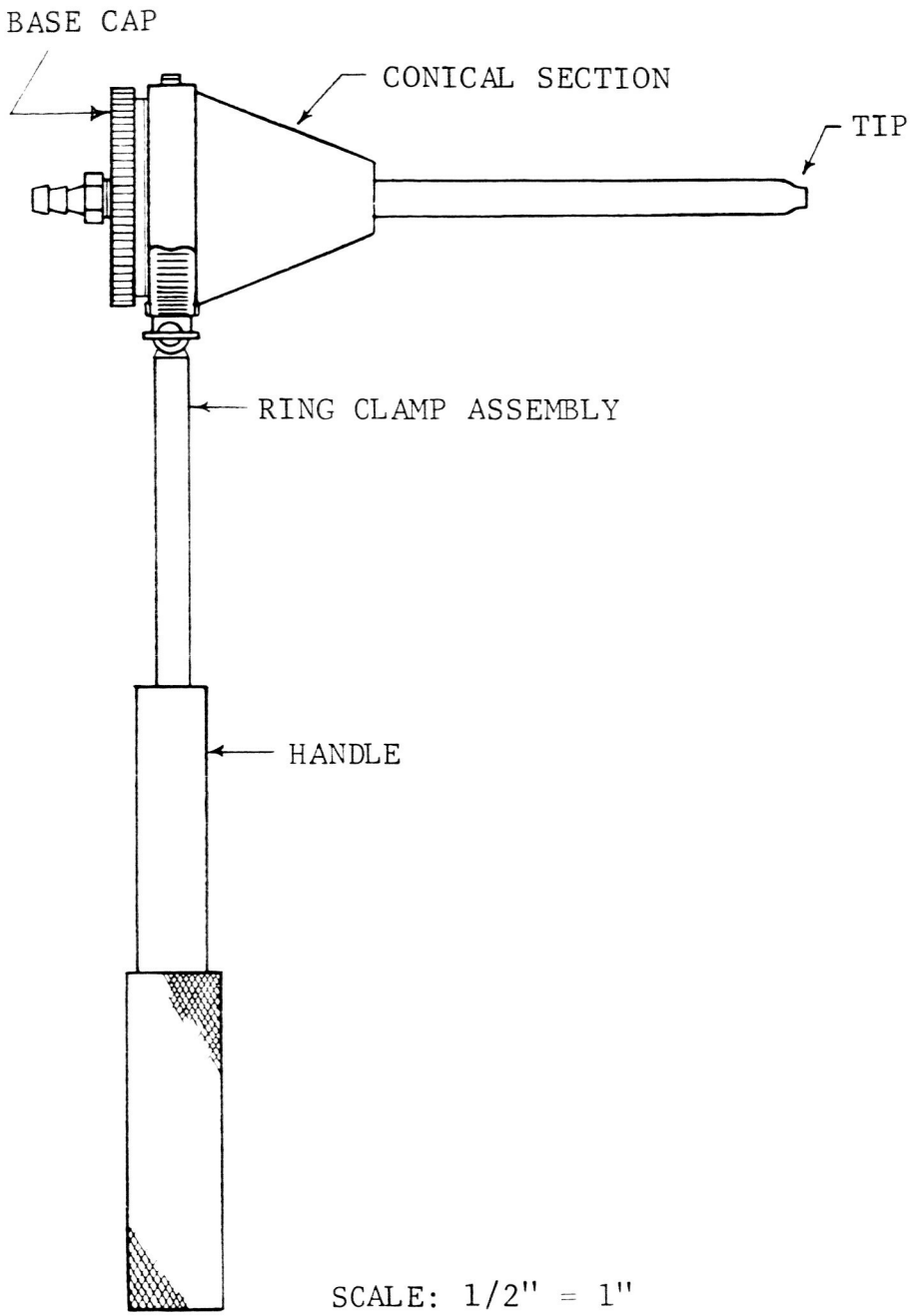



Figure 1. Filter probe assembly

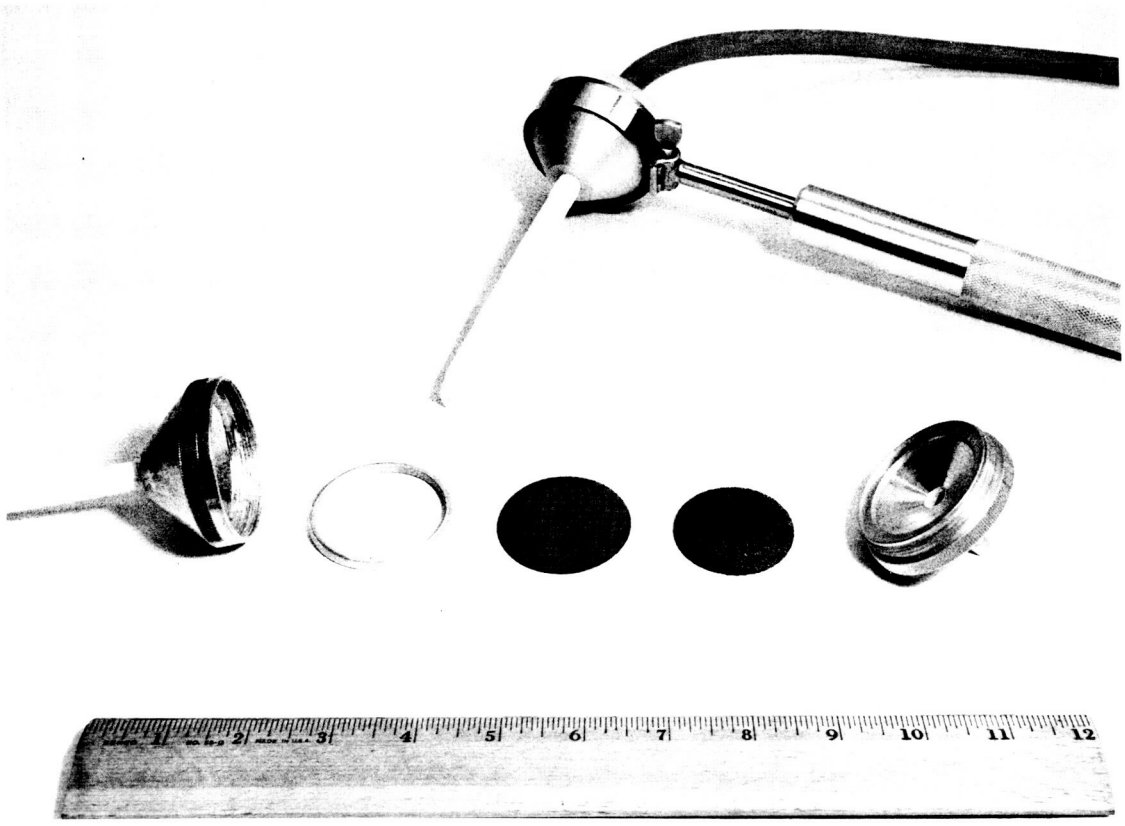
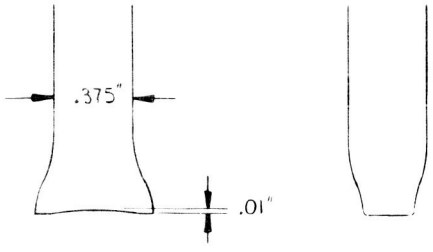
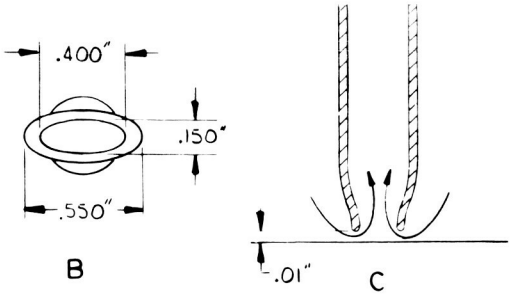


Figure 2. Disassembled and assembled models of the present filter probe




A




B C

Figure 3.
Vacuum probe schematic drawing

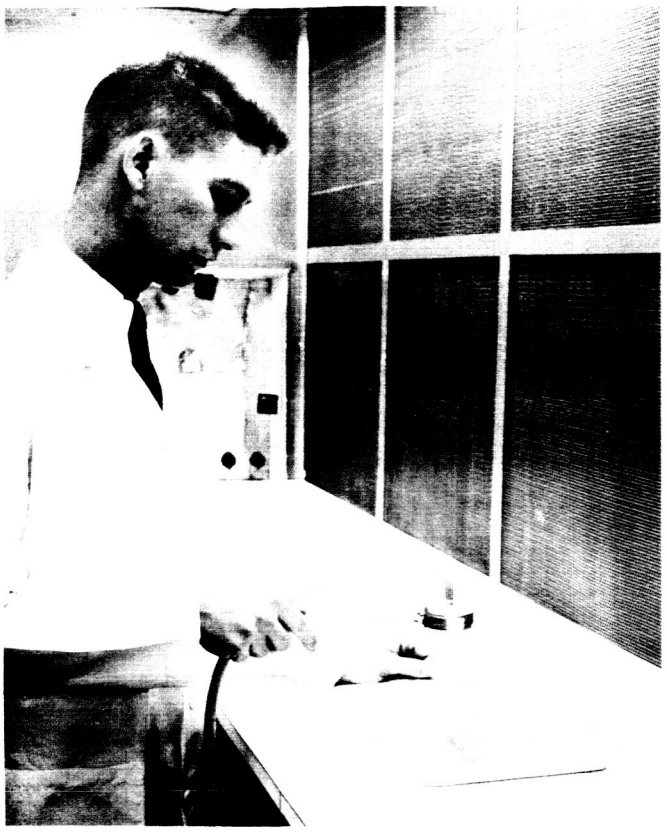


Figure 4.
In-use demonstration
of filter probe

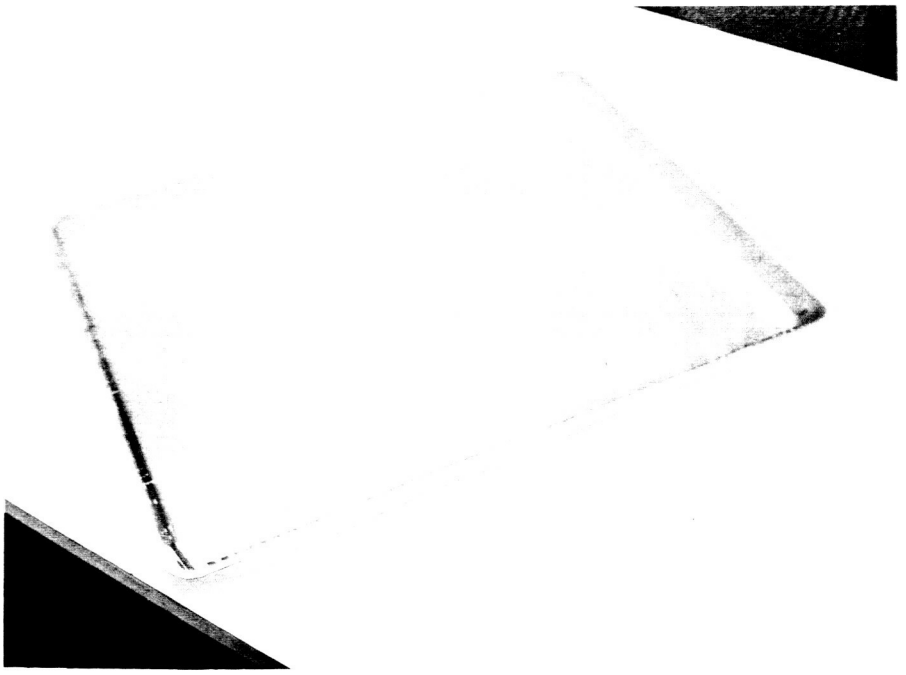


Figure 5. Covered test surface prepared for incubation

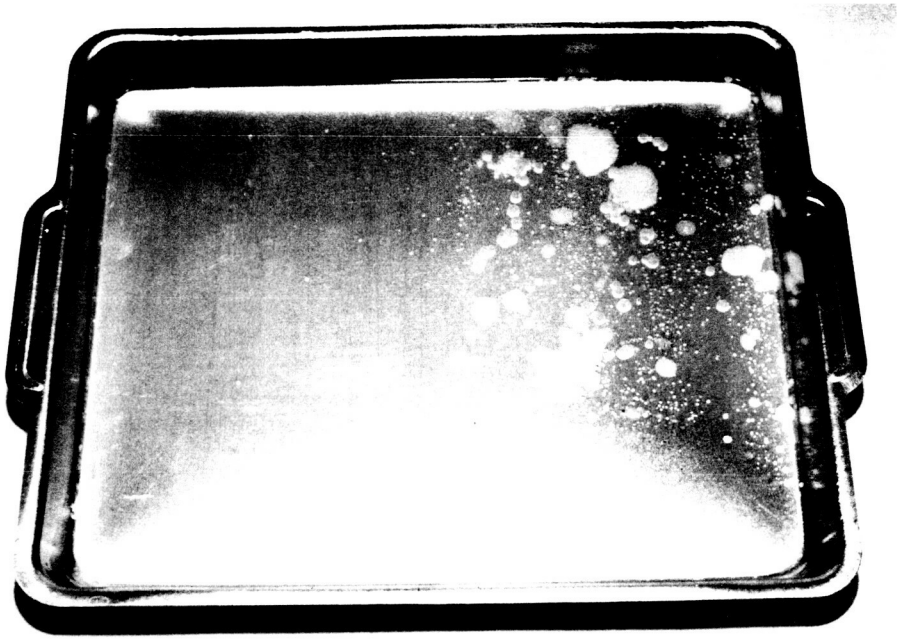


Figure 6. Appearance of vacuumed test surface and control

Figure 7. Viable particle recovery on membrane filter

also showed that the techniques used to disseminate the organisms produced a uniform loading over the test area. For the determination whether overlaying with agar disturbed or moved the particles on test surfaces, the sampled half of each pan used was first crosshatched with molten agar by using a Cornwall syringe (Figs. 8-9). After this agar solidified, the entire pan was overlaid with more agar. The crosshatching prevented the possibility of washing viable particles from the sampled half to the control half and vice versa. Essentially no microorganisms were moved by overlaying with agar.

Four different techniques were used to disseminate the viable organisms used for the experiment. First, dry spores of Bacillus subtilus var niger were disseminated by ultrasonic energy, a technique devised by V. Dugan. The assembled apparatus is shown in Fig. 10 and some of the disassembled parts are shown in Fig. 11. The results are summarized in Table 1. Second, spores of Bacillus subtilus var niger suspended in ethanol were aerosolized in a DeVilbiss No. 40 nebulizer (Fig. 12).

Figure 8. Cross-hatching test surface with Cornwall syringe

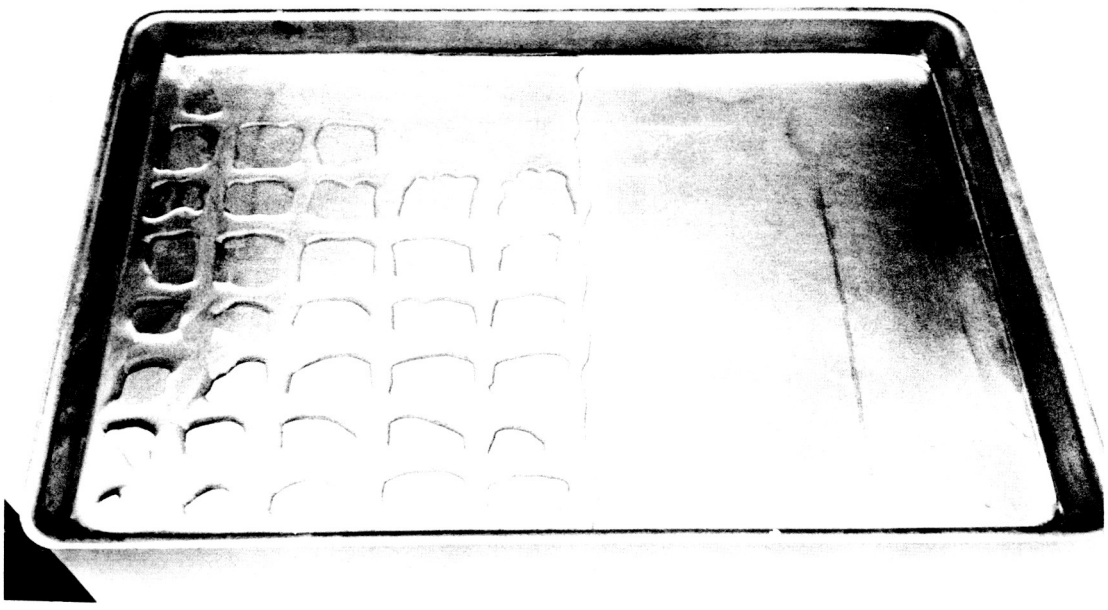


Figure 9. Appearance of cross-hatched test surface

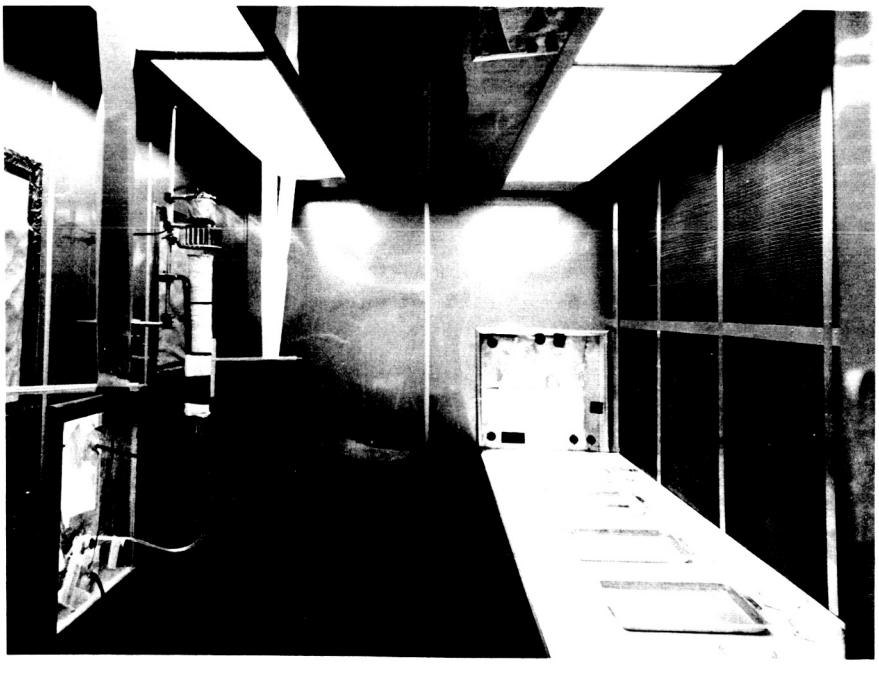


Figure 10. Experimental test chamber and test surfaces

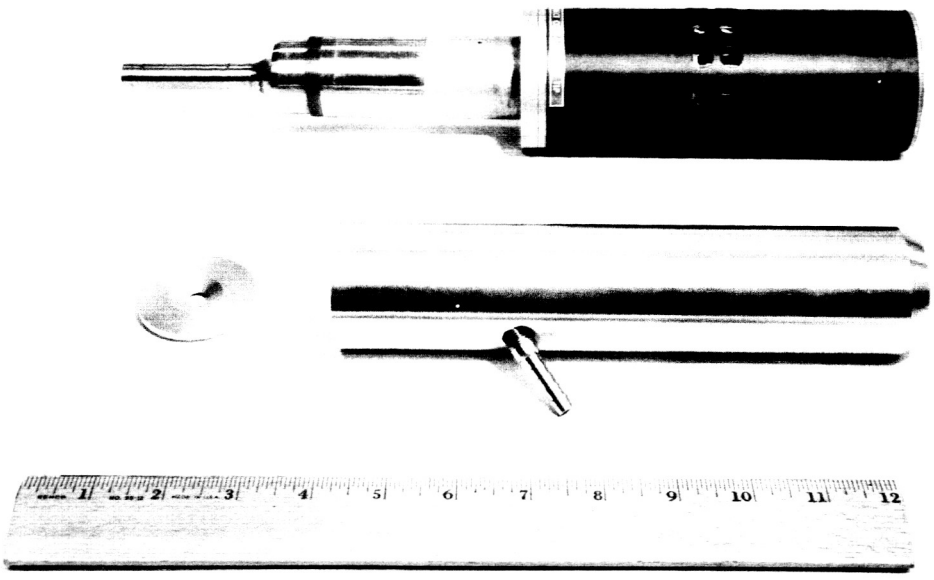


Figure 11. Sonic disseminator for aerosolization of dry bacterial spores

TABLE 1

Results of Vacuuming a Test Surface Contaminated With Dried Bacterial Spores (Bacillus subtilis var. niger)

| Probe Used | Test Surface Area | Test Surface Type | Colonies on Control Surface | Colonies on Vacuumed Surface | Percentage Removal |
|---|---|---|---|---|---|
| Stainless | 1936cm$^2$ | Alum. | 1687 | 0 | 100% |
| Stainless | 1152cm$^2$ | Alum. | 927 | 33 | 96% |
| Stainless | 968cm$^2$ | Teflon | 937 | 38 | 96% |
| Stainless | 1152cm$^2$ | Alum. | 6944 | 97 | 99% |
| Teflon | 576cm$^2$ | Alum. | 413 | 27 | 93% |
| Teflon | 576cm$^2$ | Alum. | 421 | 44 | 90% |
| Teflon | 576cm$^2$ | Alum. | 276 | 20 | 93% |
| Teflon | 576cm$^2$ | Alum. | 447 | 21 | 95% |

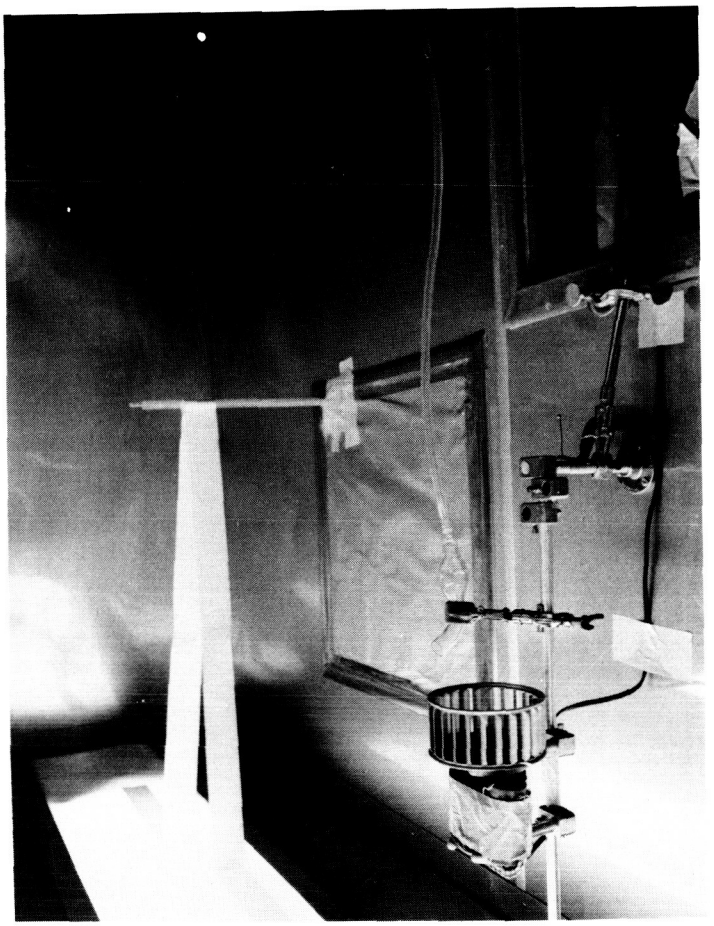


Figure 12.
Devilbiss No. 40
nebulizer

The results after using the vacuum probe are shown in Tables 2 and 3. Third, garden soil spores were disseminated by pouring garden soil from one piece of paper to another five to six feet above the test surfaces (Fig. 13). The results from vacuuming with the probe are given in Table 4. With the fourth technique, human skin flakes were disseminated by having a volunteer rub his forearms three to four feet above the test surfaces (Fig. 14). The results are given in Tables 5 and 6.

The results of very early experimentation showed that, for a high removal percentage, the volume flow rate must have reached a critical level. The condition, called a critical flow rate, is the point at which the velocity of air flow through an orifice ceases to increase with decreases in the pressure downstream from the orifice, given a constant upstream pressure (Fig. 15). An experiment (flow graph is shown in Fig. 16) was devised to show the dependence of the critical flow rate upon atmospheric pressure (Fig. 17).

Air flowing through this orifice was suspected to produce ultrasonic energy. An experiment (Fig. 18) gave a bandpass analysis of the noise generated by the tip from .89 to 44.6 kilohertz (Fig. 19). Elementary calculations showed that this noise could conceivably generate a peak force of approximately $2 \times 10^{-5}$ dynes on a micro-organism 1 micron in diameter. A considerably higher force would probably be needed to shake a 1 micron particle loose.

TABLE 2

Results of Vacuuming a Test Surface Contaminated with Wet Bacterial Spores (Bacillus subtilis var. niger)

| Probe Used | Test Surface Area | Test Surface Type | Colonies on Control Surface | Colonies on Vacuumed Surface | Percentage Removal |
|---|---|---|---|---|---|
| Stainless | 1152cm$^2$ | Alum. | 213 | 48 | 77% |
| Stainless | 1152cm$^2$ | Alum. | 1671 | 95 | 94% |
| Stainless | 576cm$^2$ | Alum. | 1004 | 181 | 82% |
| Stainless | 576cm$^2$ | Alum. | 1187 | 248 | 79% |
| Stainless | 576cm$^2$ | Alum. | 62 | 8 | 77.5% |
| Teflon | 576cm$^2$ | Alum. | 77 | 1 | 99% |
| Teflon | 576cm$^2$ | Alum. | 74 | 1 | 99% |
| Teflon | 576cm$^2$ | Alum. | 108 | 4 | 96% |
| Teflon | 576cm$^2$ | Alum. | 146 | 3 | 98% |
| Teflon | 576cm$^2$ | Alum. | 650 | 38 | 94% |

TABLE 3

Results Obtained with a Membrane Filter in Conjunction with the Vacuum Probe and B. subtilis var. niger Spores Suspended in Ethanol

| Bacteria Removed from Surface Based on Agar Overlay | Membrane Filter Colony Count | Percentage of Number Removed Counted on Filter |
|---|---|---|
| 24 | 23 | 95.8% |
| 18 | 17 | 94.4% |
| 5337 | 3038 | 56.9% |
| 6215 | 2988 | 48.1% |

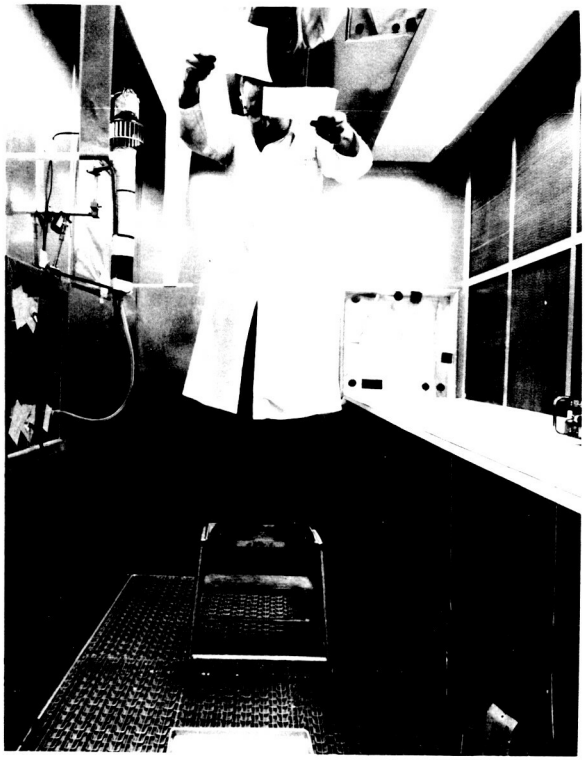


Figure 13.
Inoculation of test surfaces with soil dust

Figure 14.
Inoculation of test surfaces with skin flakes

TABLE 4

Results of Vacuuming a Test Surface Contaminated with Dust from Garden Soil

| Probe Used | Test Surface Area | Test Surface Type | Colonies on Control Surface | Colonies on Vacuumed Surface | Percentage Removal |
|---|---|---|---|---|---|
| Stainless | 1936cm$^2$ | Alum. | 2840 | 25 | 99% |
| Stainless | 1936cm$^2$ | Alum. | $1.9 \times 10^5$ | 6716 | 96% |

TABLE 5

Results of Vacuuming a Test Surface Contaminated with Human Skin Flakes

| Probe Used | Test Surface Area | Test Surface Type | Colonies on Control Surface | Colonies on Vacummed Surface | Percentage Removal |
|---|---|---|---|---|---|
| Stainless | 1152cm$^2$ | Alum. | 136 | 7 | 95% |
| Stainless | 1152cm$^2$ | Alum. | 188 | 2 | 99% |
| Teflon | 576cm$^2$ | Alum. | 233 | 6 | 97% |
| Teflon | 576cm$^2$ | Alum. | 151 | 6 | 96% |

TABLE 6

Results Obtained Using a Membrane Filter in Conjunction with the Vacuum Probe and Human Skin Flakes Contamination

| Bacteria Removed from Surface Based on Agar Overlay | Membrane Filter Colony Count | Percentage of Number Removed Counted on Filter |
|---|---|---|
| 241 | 115 | 47.7% |
| 145 | 89 | 61.4% |

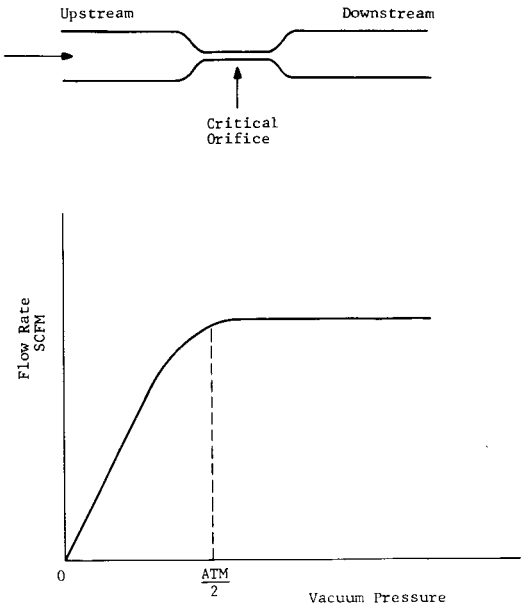



Figure 15.
Critical flow rate

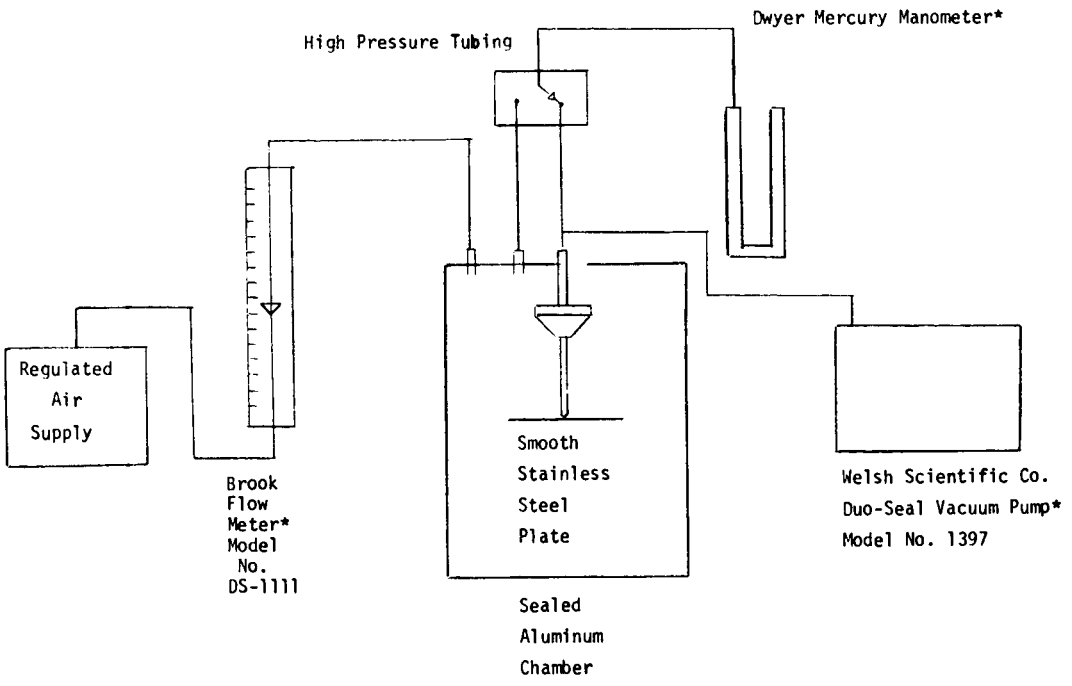



Figure 16. Test setup to determine the dependence of the vacuum probe critical orifice on operating pressures

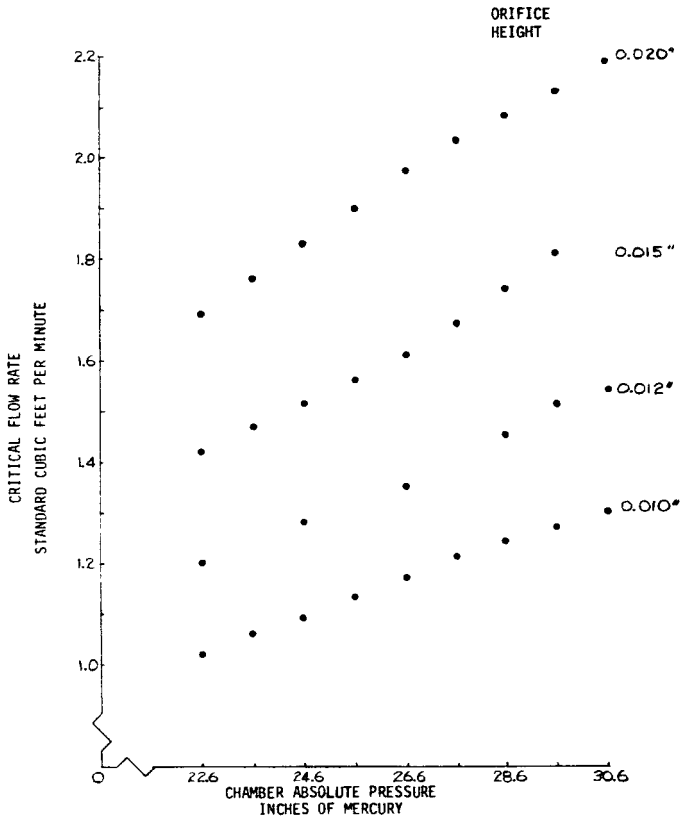



Figure 17.

Variations of critical flow with changes in atmospheric pressure for different orifice dimensions

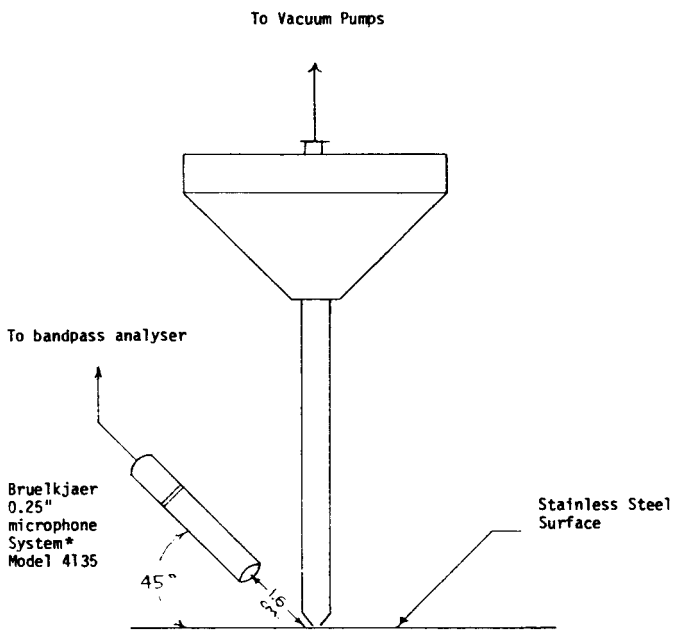



Figure 18.

Test setup for bandpass analysis of sonic noise produced by vacuum probe

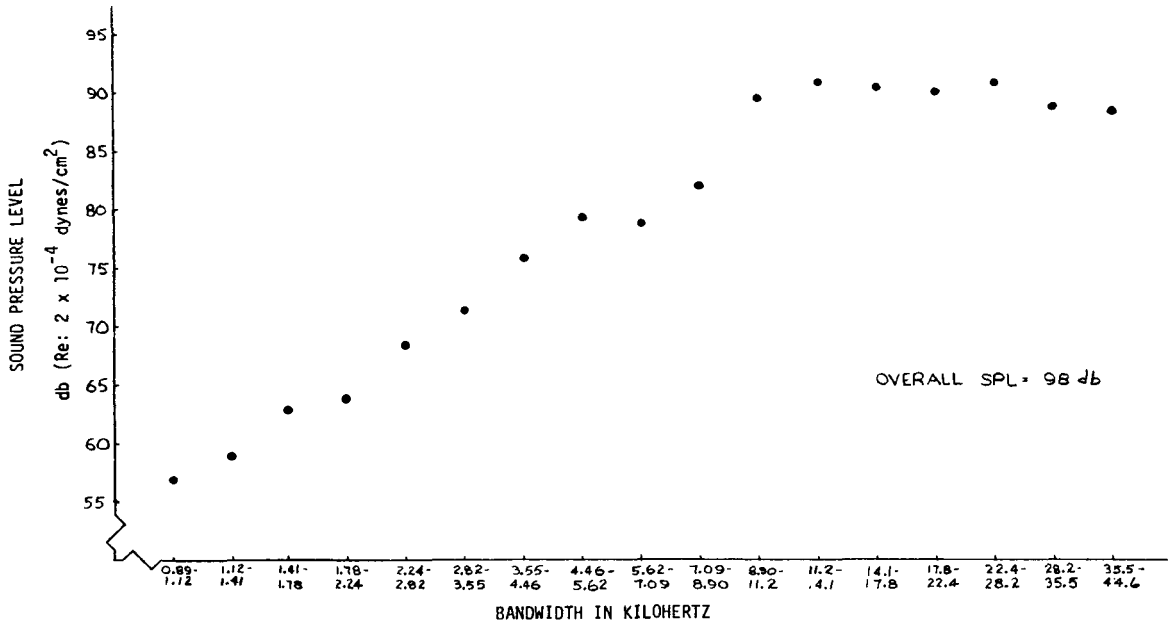



Figure 19. Bandpass analysis of sonic noise measured 1.6 centimeters from probe tip

## Summary

Testing has shown that the vacuum probe is a tool which is useful for assaying low loadings of viable micro-organisms from large surfaces within clean rooms. The reliability and repeatability exceeds that of the classical microbiological techniques adapted for this purpose.

# 4. THE PROBABILITY OF RELEASING MICRO-ORGANISMS ON FRACTURE FROM SOLIDS

by

N. J. PETERSON

U. S. Public Health Service
Phoenix, Arizona

Mathematical models are being used to predict the probability of landing a viable organism on a planet, and one of the estimated probabilities in the equations is the probability of releasing a viable micro-organism from within a solid material. The spacecraft should be sterilized so that there are no viable micro-organisms within the solid material, but this probability of release ($P_r$) still has to be considered.

The probability calculations, up to this point, has always used 1 as the worst case situation; in other words, if there is a viable micro-organism in the solid, upon impact of the spacecraft, the organism will be released. In the laboratory work on the recovery of micro-organisms from solids, we simulate impact by breaking pieces of solid material. If these have micro-organisms entrapped within them and they are released, upon impact a certain amount of the population will be killed. So we know that 1 is probably not the right number, but what is a good number?

Therefore, we decided to develop curves, using a model system, to relate concentration of spores per volume to the $P_r$ per square area exposed when fracturing takes place. We further decided to do this in as nontraumatic type approach as possible in order to do away with the generation of heat and the generation of mechanical forces which are responsible for the die-off when solid pieces of material are fractured.

The model system is a polymerized plastic, with leucite as the main ingredient. Spores can be entrapped in the plastic, within which we can predict the level of contamination in terms of spores per gram or spores per cubic centimeter. We have the ability to sample and to check the prediction of this concentration because leucite is soluble in acetone, and acetone is not lethal to the spores. By dissolving the plastic and filtering it, we can tell exactly how many spores there are per unit volume.

Batches of the plastic are cut into small discs 14 millimeters in diameter and 2 millimeters thick. By breaking these discs across one diameter, two surfaces will be exposed, each surface being 2 x 14 millimeters and exposing 28 millimeters squared. These halves can be broken again along the quarter plane to expose another 28 millimeters. After breaking these in various ways (being sure that the surfaces are sterilized), we drop these pieces with exposed surfaces into tubes of broth. We incubate the broth for a minimum of 30 days and watch for growth. If growth occurs, we say that at least one micro-organism was released or was exposed at the surface, and if no growth occurs, we say that no micro-organisms were released at the surface. If we have run 20 tubes,

we can say that the number of tubes that turned positive represents a close approximation of the probability of at least the number of spores released per unit per square area exposed. We then plotted the points and fitted a line to them for four surface areas, so we now have 4 lines in which we relate the $P_r$ versus concentration in spores per gram or spores per cubic centimeter. This is useful in one respect because it gives a much better estimation than we had before.

A second thing done with these curves is picking off points and transferring them to another graph for extrapolation. Now we plot surface area exposed versus $P_r$ and get a family of curves for the various concentrations of spores within the plastic. This allows us to find at what surface area exposed the $P_r$ will be. We have tried to get a better approximation of $P_r$, and we have found out that it is not Number 1. For example, we have never found a positive surface at anything below 500 spores per gram. This is still quite contaminated, and we expect the spacecraft to be considerably less contaminated than that. Below that, we have all zeros, so $P_r$ is definitely not 1, especially since the impact and traumatic fracturing is going to be more destructive than the simple fracturing done in the laboratory. This work will be followed up to see whether it does bear some good relationship to what happens under impact conditions.

# 5. LIFE DETECTION EXPERIMENTS

by

G. L. HOBBY

Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California

## Introduction

Since 1960, NASA has supported the development of life detection experiments for unmanned planetary probes to Mars. These experiments have consisted mainly of schemes for testing soil samples to determine whether they contain organic compounds of biological origin, or whether they exhibit any active processes, such as metabolism, photosynthesis, or replication. Three of these experiments are discussed below.

## Gas Chromatography-Mass Spectrometry

Both gas chromatography and mass spectrometry are powerful chemical analytical tools. In the last few years, it has been possible to combine these two instruments into a single system by using gas chromatography for separating volatile components and mass spectrometry for identifying them. This technique became possible when molecular separators were developed for separating the carrier gas effluent from component samples before these components were injected into the ion source of the mass spectrometer. By combining this method with computer analysis of the mass spectra, rapid analyses of mixtures of volatile organic compounds are possible.

Such an instrument system has been proposed for application to planetary exploration and it is being developed by the Jet Propulsion Laboratory under NASA sponsorship. One of its most attractive features is its great versatility. It can be used to perform extensive analyses of organic surface material, it can determine the gaseous composition of the planet's atmosphere, and it also can be used in experiments that test for metabolic processes.

For the analysis of nonvolatile organic compounds in Martian soil with the gas chromatograph-mass spectrometer system, these compounds must be converted into volatile forms that are characteristic of the chemical classes in the starting sample. One of the important projects that NASA is supporting at JPL is to determine what techniques of pyrolysis will convert complex organic mixtures in soil to volatile organic products that are identifiable with the different compounds in the

---

This paper presents the results of one phase of research carried out at the Jet Propulsion Laboratory under Contract No. NAS7-100, sponsored by NASA.

original mixture. Pyrolysis is of particular interest as a method of volatilizing samples for analyses by gas-chromatography-mass spectrometry on planetary probes because it is simpler than methods involving wet chemical processes, as, for example, preparing volatile derivatives of components in the sample. In addition, another advantage is that samples consisting of insoluble organic matter can be analyzed.

Experience has already been gained in designing gas chromatograph and mass spectrometer instrumentation for space probes. Mass spectrometers have been flown on sounding rockets for analyses of the Earth's upper atmosphere. An instrument for planetary space probes will be much more sophisticated. Figure 1 illustrates the basic configuration of a gas chromatograph-mass spectrometer system for planetary use. The essential parts are the carrier gas storage tank, the pyrolysis oven, the gas chromatographic column and detector, the molecular separator, and the mass spectrometer. For an organic analyses, a sample of soil is obtained from the surface by a sample collection system, and a portion is dumped into the pyrolysis oven. After or during pyrolysis any volatile organic products are swept from the oven into the gas chromatograph column for separation. As each component passes the chromatograph detector, the mass spectrometer begins scanning. Excess carrier gas is removed from the system by the molecular separator, and the concentrated sample is injected directly into the ion source of the mass spectrometer. The signal output from both the gas chromatograph and mass spectrometer is fed into an electronic data system for processing in order to compress the total data before it is transmitted to Earth.

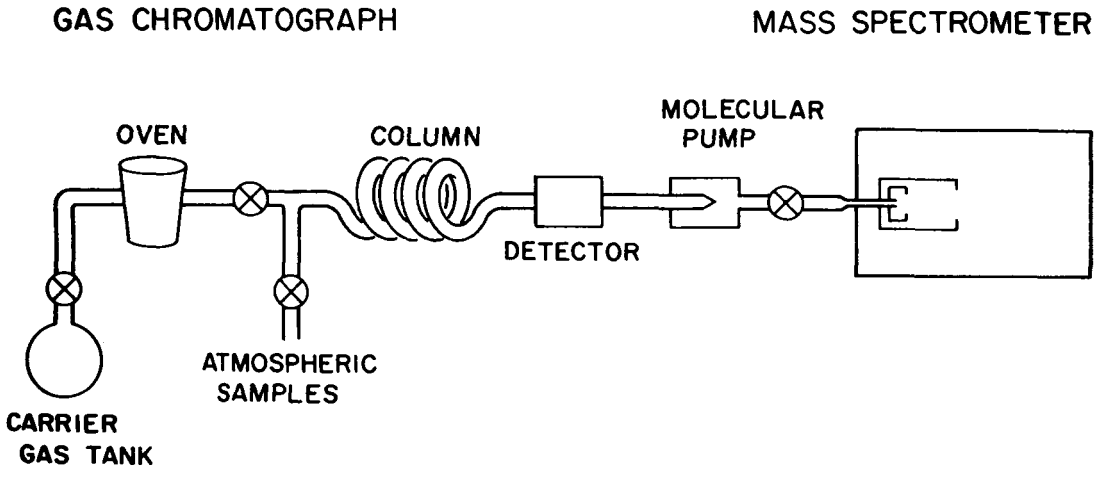



Figure 1. Basic GC-MS experiment

Figure 2 is an artistic conception of the instrument for use on a planetary probe. The component parts include the hopper for receiving a sample, the pyrolysis oven, the carrier-gas tank, the columns and detectors, the molecular separator and pump for removing carrier gas, the ion source of the mass spectrometer, the electrostatic analyzer, the magnet for the ion pump for evacuation of the MS, the magnetic analyzer and the low and high mass sectors containing the electron multipliers.

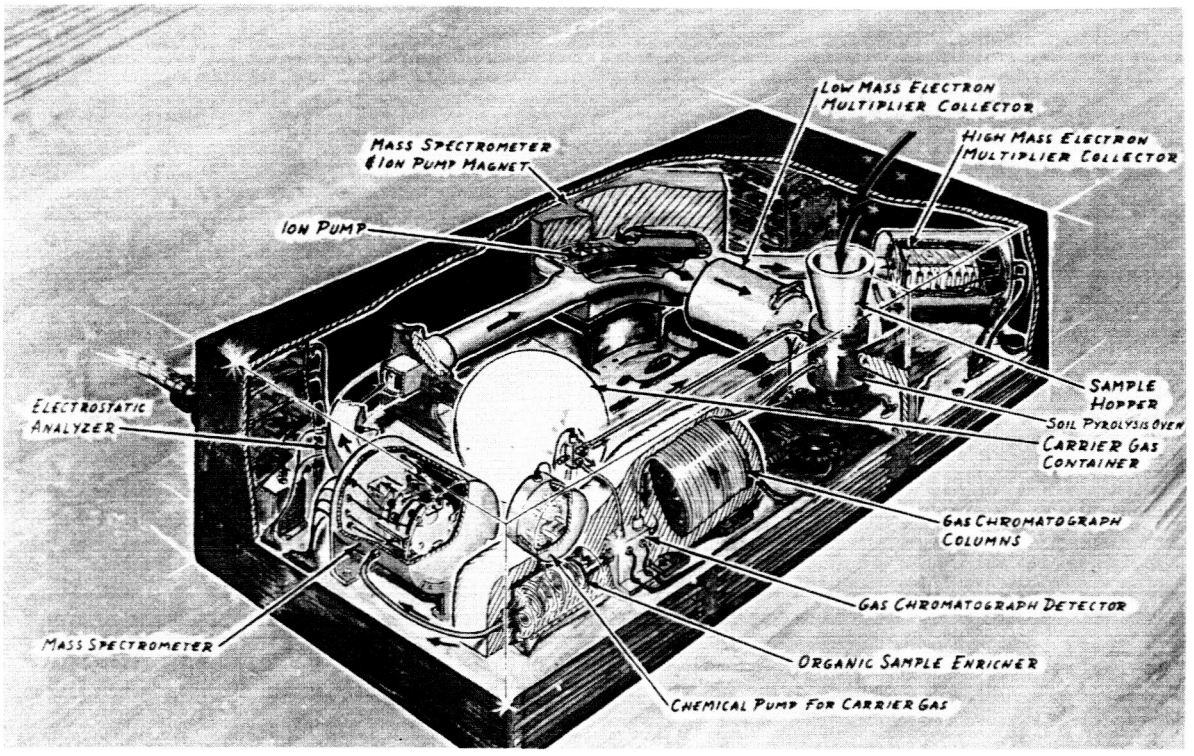



Figure 2. Gas chromatograph mass spectrometer for Mars soil organic analysis

A complete flight instrument, including all electronics, would weigh about 30 pounds, and consume an average of 20 watts of power, (50 watts for seconds or minutes during pyrolysis). It would cover a mass range of 1 to 350 at unit mass resolution, have a 7 second scan time, and a dynamic range of $10^6$.

Figure 3 shows an engineering model of a compact gas chromatograph that was designed exclusively for atmospheric analyses. This illustrates what can be done in designing instruments for special space applications. This gas chromatograph can withstand shocks of 3,000 g's. Components that are visible in this view are the pressure regulator valve, the thermal conductivity detectors and four short stainless steel columns. The instrument is 7 x 7-1/2 x 4 inches in size and weighs about 10 pounds.

Figure 4 shows a breadboard model of a mass spectrometer. This is a single sector, double-focusing device with a mass range of 100, and unit resolution. The main components shown are the ion source, the electrostatic analyzer, the magnetic analyzer, the collector slit and the mounting for the electron multiplier. The weight of this instrument is about 7 pounds.

The following two experiments deal with the problem of searching for evidence of physiological processes in samples of Martian soil.

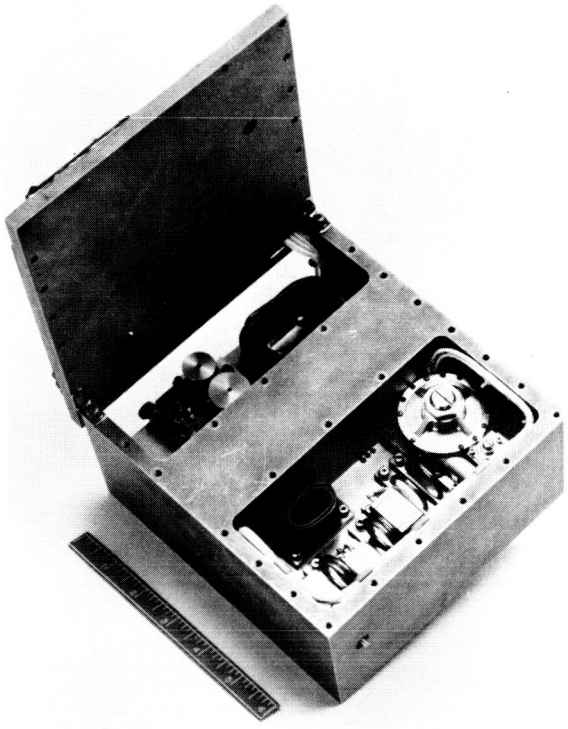


Figure 3. Gas chromatograph for atmospheric analyses

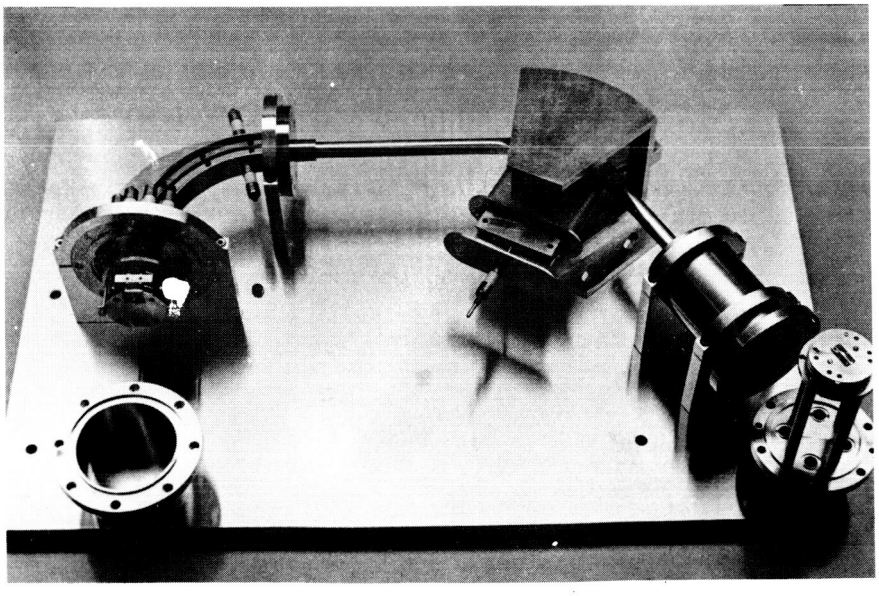


Figure 4. Breadboard model of mass spectrometer

## Detection of Carbon Dioxide Assimilation

The first of these is a simple approach for detecting biological carbon dioxide fixation. Soil samples are incubated in chambers containing an atmosphere of carbon dioxide labelled with C-14. The samples are incubated both under natural Martian illumination and in complete darkness for periods of hours or days. Replicate samples sterilized either by heat or with chemicals are also incubated. After incubation, each sample is treated with acid to remove adsorbed $CO_2$, or accumulated carbonates, and excess radioactive carbon dioxide is purged from the chamber. The soil samples are dried and the accumulated radioactivity measured by placing a radiation detector directly over the sample. Differences in radioactivity between the illuminated, the darkened, and the sterile samples will indicate whether carbon-14 has been assimilated. As might be expected, this technique works very well for most samples of terrestrial soils. Table 1 shows typical results obtained when as little as 50 mgs of soil are incubated. Dark $CO_2$ fixation as well as photosynthetic activity is commonly observed.

Figure 5 shows how these soils are incubated in the laboratory. No medium is used in testing the samples. They are simply moistened with distilled water before they are incubated.

The planchet counting method is inherently inefficient because of the poor counting geometry and self-absorption by the soil samples. The radioactivity measurements of soils spread on planchets are less than five percent efficient. An alternative scheme is illustrated in Figure 6. After incubation the soil samples will be pyrolyzed under conditions that will produce organic pyrolysis products. Carbon dioxide, water, and carbon monoxide that may form will be separated from organic pyrolysis products by gas chromatography, and the total volatile organic fraction will be collected in an internal gas radiation detector. Radioactive organic pyrolysis products obtained from carbon-14 labelled organic substrates produced during incubation will indicate the assimilation of carbon dioxide. Another alternative is combusting the sample completely to $CO_2$ and water, separating the water from the carbon dioxide by gas chromatography and filling the radiation detector with the carbon dioxide, some of which will be radioactive if C-14 has been assimilated.

The Gulliver experiment, developed by Dr. Gilbert Levin, formerly of the Hazeltine Laboratories, and Prof. N. Horowitz, of the California Institute of Technology, is designed to search for heterotrophic metabolism in Martian soil samples. The experiment consists of inoculating a nutrient broth containing one or more simple carbon compounds that are labelled with carbon-14. If Martian organisms are capable of metabolizing these constituents, they may form labelled carbon dioxide as a metabolic end-product. The radioactive carbon dioxide released is collected periodically and the rate of change of radioactivity is determined. If this change has a logarithmic character, it will suggest a growing culture. If the increase is linear, it will indicate either a metabolizing culture at rest, or perhaps an artifact, due to a non-biological interaction of the sample with the constituents of the medium. For this reason, a sterile control is essential in the experiment.

The basic Gulliver device is illustrated in Fig. 7. Samples of surface matter are collected by deploying a sticky string onto the surface. This is done by a small projectile which is shot away from the spacecraft. The other end of the string is attached to a drum inside

TABLE 1

Growth and Photosynthesis
Photosynthesis in JPL Soil Samples

| Counts per minute - corrected | | | |
|---|---|---|---|
| Light | | Dark | |
| Unheated | Autoclaved | Unheated | Autoclaved |
| 3114 | 67 | 83 | 54 |
| 2396 | 162 | 123 | 84 |
| 2304 | | 112 | |
| 3710 | | 83 | |

| | |
|---|---|
| Sample size: | 50 mg |
| $^{14}CO_2$ | 0.18 $\mu$C/ml |
| Incubation: | 5 hr |
| Acidification: | 0.04 ml CONC HCL |
| Purge Time (Air): | 30 min |
| Illumination: | 750 fc |
| Temperature: | 26°C |

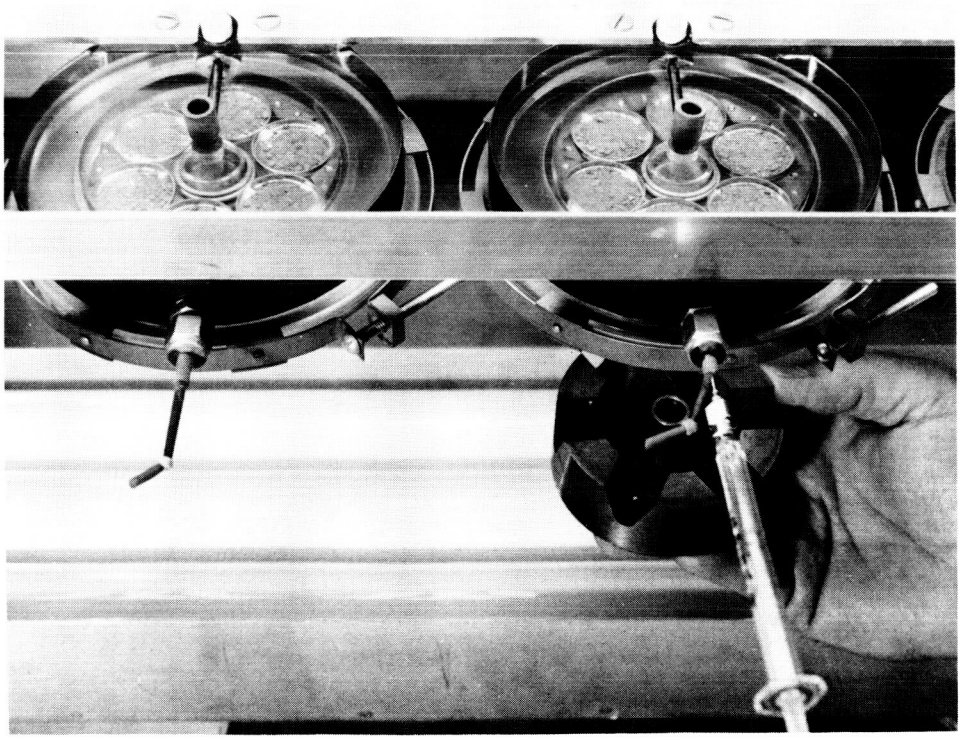


Figure 5. Incubation of soils

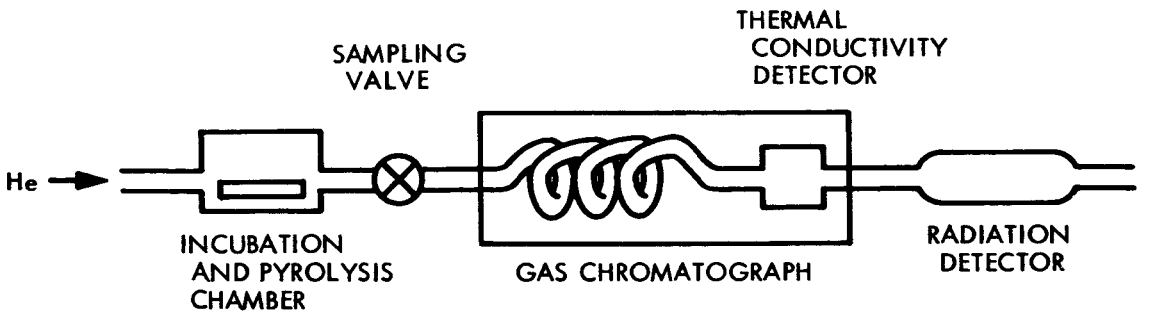



Figure 6. Photosynthetic life detection system

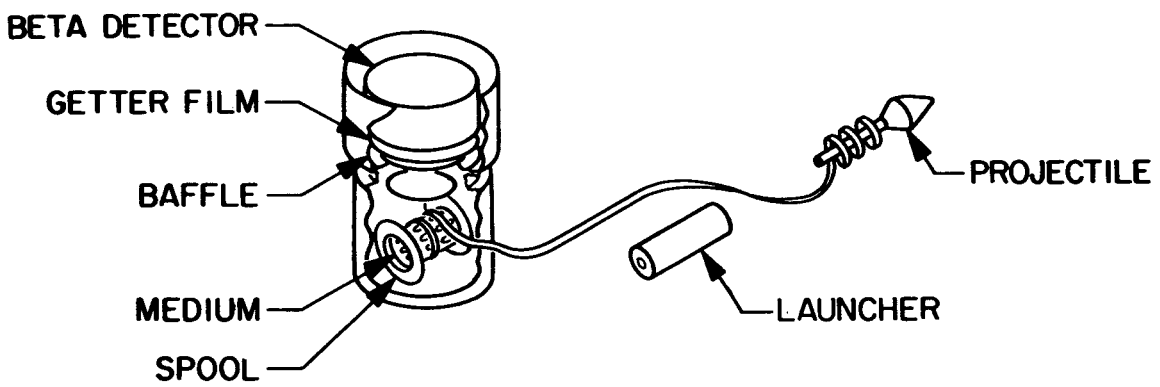



Figure 7. Basic Gulliver experiment

the growth chamber, and after deployment, the drum rotates and pulls the string across the soil surface and into the chamber. Soil particles and micro-organisms are picked-up by the string. The hub of the drum is perforated and is hollow. Inside the hub there is a vial of growth medium. After the string has been completely wound on the drum, the chamber is closed and the vial of growth medium is punctured by a plunger. The growth medium is adsorbed by the string and saturates the adhering soil particles. If the radioactive substrates are metabolized to carbon dioxide, this radioactive gas is released and will diffuse past a set of baffles to a thin layer of barium hydroxide deposited on the window of the Geiger Muller tube.

A model 3 prototype of this system has been successfully tested in a variety of locations, such as mountain peaks and dry desert regions. This instrument is shown in Fig. 8. The two projectiles for deploying the sampling strings are in the foreground. The growth chamber is in the center. The two ports for the strings are at the top of the chamber, and the top structure houses the Geiger tube and anti-coincidence electronics that compensate for background radiation.

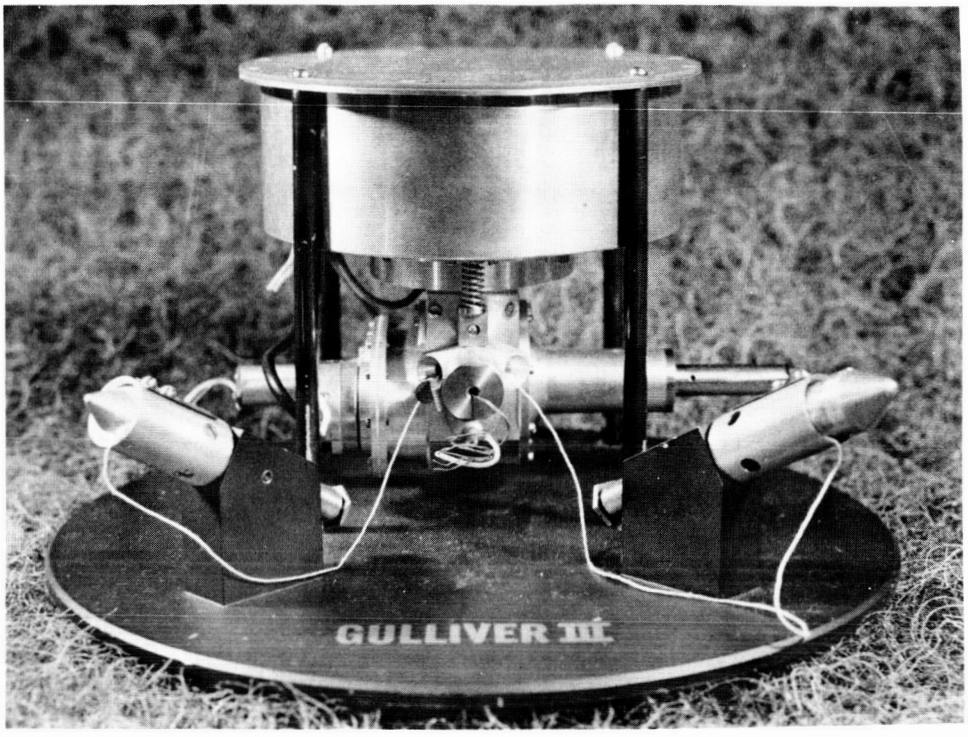



Figure 8. The Gulliver III

Figure 9 is a cut-away view of the instrument. The broth a le is in the center of the spool, and the ampoule breaker is in the i- zontal cylindrical structure on the right side of the instrument. The ampoule containing the sterilizing agent and the breaker mechanism for shattering the ampoule is to the left.

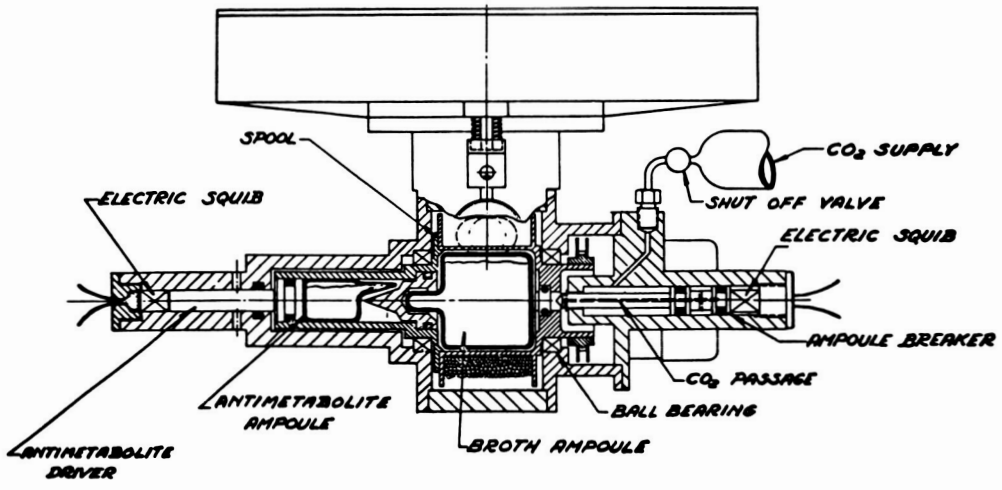



Figure 9. Side elevation, cutaway of Gulliver III

Figure 10 shows the instrument mounted in a small capsule in preparation for a field test. Figure 11 shows the strings being drawn into the instrument during one of the field trials at a desert test site.

Figure 12 shows results of one of the tests performed near Salton Sea, California. Note that in the first 30 minutes a very rapid response is obtained, after which only a very slow metabolic rate is observed. This is typical of most trials, either when using this model instrument or in laboratory testing of soils.

Several other approaches are being developed. These include the detection of growth of micro-organisms in liquid suspensions of soil particles by changes in the forward scattering of light (Wolf trap); the detection of nucleic acid, proteins, and porphyrins by fluorometry; fluorometric assays for enzymes; microscopy; and the analyses of soil organic matter by infrared spectrophotometry.

In summary, a gas chromatrograph-mass spectrometer system appears to be one of the most versatile tools for obtaining information about the organic chemistry and the biochemistry of the Martian surface. It is also ideal for determining accurately the composition of the Martian atmosphere.

Pyrolysis is the simplest method of preparing nonvolatile organic samples for a gas chromatograph-mass spectrometer analyses on planetary probes. The technology of miniaturizing these instruments is relatively advanced.

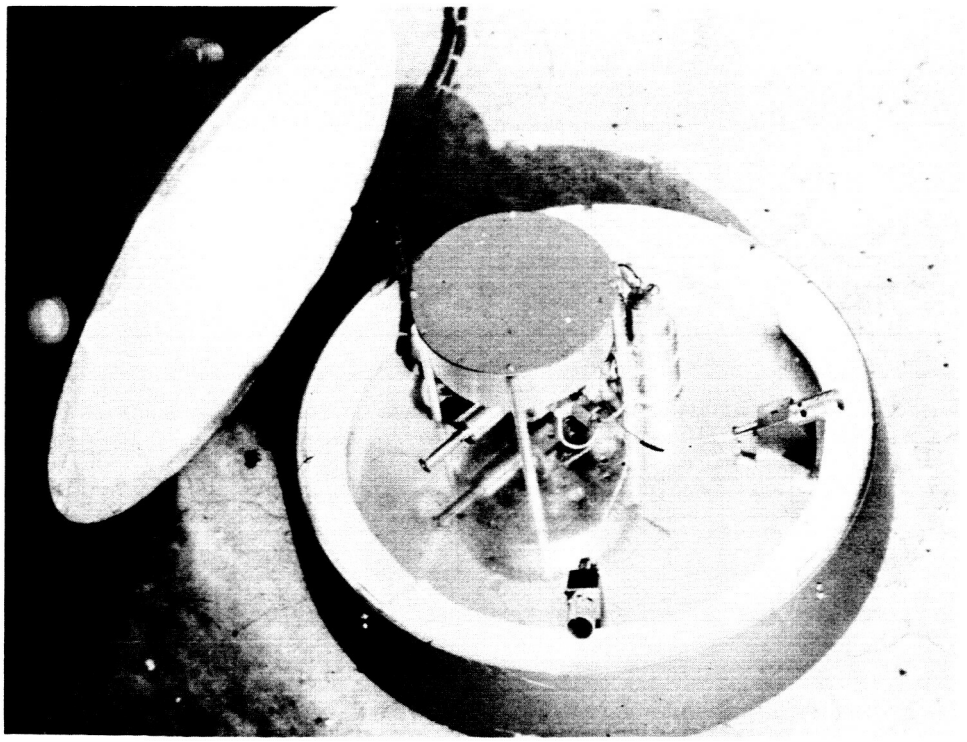


Figure 10. Gulliver III mounted in capsule

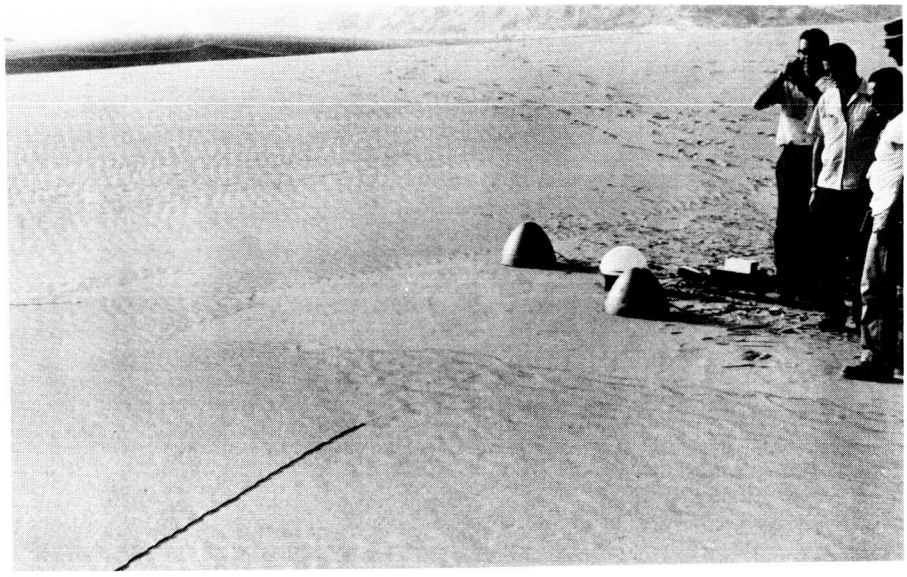


Figure 11. Field trial of Gulliver III, and desert test site

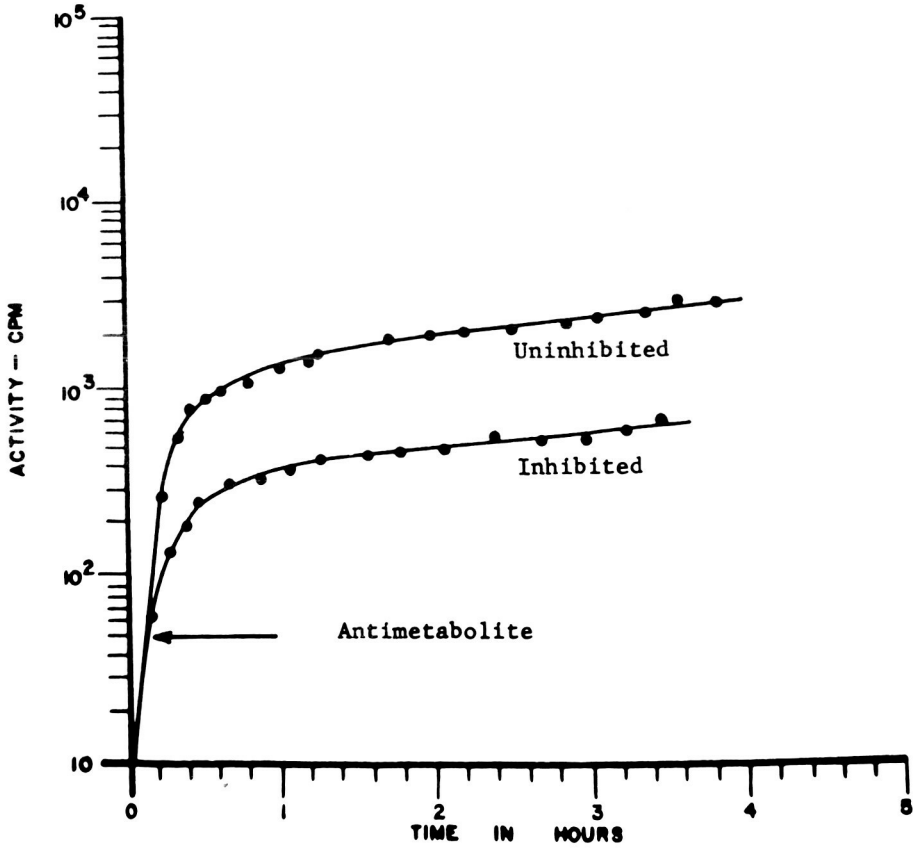



Figure 12. Field test response - first five hours (Salton Sea, California)

Experiments for detecting physiological activity in soil samples are also being developed and some of these have been tested in the laboratory and in the field.

The combined data from these, and other experiments, being developed by NASA should provide a reasonably clear picture of whether life is, or is not, present on Mars.

Question and Answer Period

OGLE: Was the spectroanalyzed acetone or other fluids checked for particulate content?

LeDOUX: No, not previous to their use. The spectroanalyzed acetone is triple distilled. This is used directly on the spacecraft, not for the killing of organisms, but primarily for their physical removal. The isopropyl alcohol was 200 proof, and it was cut down 10 percent to 180 proof. It is filtered through membranes and filtration is obtained down to 5/10 micron in size. We found that the spores were washed free from the component, and in order not to contaminate other components that may not have spore forms on them, we filtered every time that we used the isopropyl alcohol.

FEY: Did I understand you to say that, after spending a certain amount of time in outer space environment, the only remaining viable microbes would be those inside electronic components such as transistors? Do you have a program for decontaminating and monitoring such components.

LeDOUX: Yes, I did say that. And probably the only forms there would be spore forms. Vegetative life would no longer exist after going through 1,440 cyclic changes between -45°C and +50°C. The exterior of transistors and other electronic components will be washed in alcohol for 15 minutes. However, the interiors will not be decontaminated. During manufacture, these components exceed 1000°C, so we feel reasonably sure that most of these do not contain viable life within them. There are about 37,000 components in the spacecraft, and the assessment is based on that.

YEICH: Is a plan being formulated to check for extraterrestial bacteria in returning spacecraft before reentry? Will agar plates be exposed and studied by the astronauts?

FAVERO: The astronauts are not going to do any biological tests on the surface of the moon. The lunar material will be delivered to the Lunar Receiving Laboratory (LRL) at Houston, and here the samples will be safety tested. Part of the safety testing is determining whether or not there are any organisms or any toxic material in the lunar sample.

YEICH: Astronauts returning from extraterrestial landings will be quarantined. How will the spacecraft be decontaminated since we do not know what type of life may be brought back? How can we be protected from organisms in and on the spacecraft escaping after reentry?

FAVERO: The outside of the capsule probably would not get contaminated, although there is the possibility because this is the ship that will be orbiting the moon. The module that goes to and returns from the surface of the moon to this orbiting ship will not be brought back to earth. The heat of reentry on the surface of the returning capsule probably would kill any viable material, as we know it. With respect to the interior of the capsule, the primary rationale is not mainly decontamination, but containment. The astronauts, the samples, and other materials will be contained within LRL until they are safety

tested. This is extremely important because there might be organisms in the layers of the lunar surface. The probability of lunar life is very low, but if it were to exist, the penalties would be extremely high, especially with viable and toxic materials.

YEICH: What about the aspect of unknown types of organisms.

FAVERO: We do not know whether decontamination techniques, such as heat and chemicals, affect organisms with a different make-up. The only thing we can do now is to work up materials, methods, and applications according to how we understand life on this planet.

NEILL: Let me enlarge on the LRL facility. It is on final check-out now at Houston. It is an $8,500,000 building and is equipped with all kinds of modern instrumentation and assay techniques; at present, there are about 50 full-time employees. All of the safety tests that will be performed on the returned samples, which amount to about 50 lbs, will be within thick containment barriers. The minimum amount of lunar material for quarantine purposes to determine whether there is anything that will affect people or other living things on this earth is about 1-1/2 kilograms. A certain number of tests are mandatory in accordance with Federal statutory requirements of agencies such as the Department of the Interior for animal life, the Department of Agriculture for animal life and vegetation, and Public Health Service for foreign quarantine, in this case, planetary quarantine. So there are very elaborate preparations being made with respect to lunar samples to protect all of us.

SHAW: How do you sterilize saran wrap?

MORRIS: The saran wrap is already sterile as-received. However, we do have to be careful that we do not contact the surface to be used with hands or anything else that is contaminated. That saran wrap is sterile is well documented, and we have never observed an outgrowth in any of the cultures when saran wrap was used to cover them. Saran wrap has been used in tissue culture and virus studies without additional sterilization.

SHAW: What are the physiological effects of 25-40 kc ultrasonics as compared to sonic frequencies?

MORRIS: We do not know of any definite experimentation in this area except by Mr. Peterson, who spoke earlier. He has performed some tests with ultrasonic equipment for agitation of microbes and found that there was negligible kill-off. Another person has developed a model which demonstrates that organisms could conceivably be dislodged by these frequencies, but we are not too certain about that.

BALLARD: Have you experimented with a light scattering photometer hooked up with the vacuum probe to count and/or size the bacterial pickup?

MORRIS: No, but a study will be made as soon as the high rate particle sampler is developed. We would like to remove the filter from the probe and put a higher particle rate sampler directly behind it. This would simplify counting numbers of micro-organisms and particles removed from surfaces.

TRAUTH: Mr. Peterson, are you not really discussing $P_r$ from fracture rather than a general $P_r$? For example, it is not possible, as far as I know, to assert on the basis of experimental evidence that there are not life forms on Mars that make release from solids a certainty.

PETERSON: Yes, the $P_r$ referred to was that resulting from fracture. We were not talking about the release that may result from degradation of solids because of chemical or biological life action.

TRAUTH: Have you developed a model of fracture and are you using your data to verify it? Are the data themselves the model? Your reference to $P_r$ being proportional to the area after fracture prompts this question.

PETERSON: We did not develop a mathematical model. We felt intuitively that an increase in the area of any given volume of material will increase $P_r$. We took an empirical approach and the data involve many assumptions and many restrictions. We are dealing strictly with a nonporous solid and a nontraumatic type of fracture. It should be emphasized again that this is a shaky first step and by no means a definitive approach to the problem of $P_r$.

TRAUTH: What limitation do the GC-MS and other experiments have with regard to spacecraft sterilization?

HOBBY: The present components for the GC-MS have not been tested, as far as we know, under sterilization environment, but we would expect that they would survive because of the materials used to build the instruments. On the other experiments, Gulliver has gone through a sterilization cycle at least once. One problem is with the growth medium. Although the growth medium was able to support growth after the sterilization cycle, there were slight changes in coloration. One of the concerns is that we may know less about the physiology of margin organisms when the constitution of the media is changed by heating. In other words, there may be a component in the medium that margin organisms do not like, but after it is heated, they do like it, or vice versa. As far as the photosynthetic study is concerned, we have not gotten into the instrumentation phase yet.

TRAUTH: You refer to identification of "complex" organic molecules. Is it possible to identify something as complex as DNA with GC-MS techniques?

HOBBY: There is every reason to expect that we can. There is a great deal of work recorded in the literature on the analysis of polymers, and for every kind of polymer, a characteristic chromotographic pattern can be obtained. We are not sure about DNA, but things such as proteins and porphyrins give characteristic breakdown products which enable one to identify the original compound. The problem at JPL in analyzing the soils is that the soils are a mixture of complicated molecules. At present, we believe that there will be a number of sets of conditions under which we can finalize and extract sufficient information from the kinds and amounts of pyrolysis products so that we will be able to piece together most of the things that were in the original sample.

SHAW: Are you considering the possibility of any system of life other than that based on carbon?

SESSION VI

SYSTEMS APPROACH

Presentation

A SYSTEMS APPROACH TO CONTAMINATION CONTROL -- C. A. Trauth, Jr.

Session Chairman

H. D. SIVINSKI
Sandia Corporation
Albuquerque, New Mexico

HOBBY: No. From what we know about chemistry in general, one could speculate that there may be a silicon base life or a life which uses ammonia as a solvent rather than water. But here we cannot piece together a complete system to test experimentally. Most of us feel that we would be wasting time trying to look for an exotic form of life on Mars. The first thing to do is to look for the kinds of forms that have chemistry and the kinds of functions with which we are familiar.

SESSION VI

SYSTEMS APPROACH

# A SYSTEMS APPROACH TO CONTAMINATION CONTROL

by

C. A. TRAUTH, JR.

Systems Studies Division
Sandia Corporation
Albuquerque, New Mexico

Abstract

Contamination control, in spite of its increasing importance in our technically advanced society, is still a subject suffering from a lack of cohesiveness. This situation obtains because there is no theory of contamination control which applies to all specific contamination control problems and encompasses all types of contamination control techniques. This paper represents an attempt to formulate a framework in which such a theory may be developed. In effect, this is a framework in which contamination control may be planned for on a cost-effectiveness basis.

## Introduction

Contamination Control. When contamination control is understood in the broad sense of limiting or removing unwanted material, nearly every human being is involved, to some degree, in this activity. This participation may take the form of placing trash in receptacles or merely limiting one's food to things which are hoped not to be harmful. Because of this "universality" of contamination control, it is easy to view the field as a disjointed collection of relatively unrelated problems. A little reflection, however, must lead to the realization that nearly all contamination control problems have certain similar features: they involve limiting or removing particulate matter, gases or liquids in, on or from solids, gases or liquids.[1] The following abbreviated list gives some indication of the "universality" of contamination control and the similarity of the problems arising in the field.

[1] Contamination control has been viewed in some detail in this form in a document entitled Principles of Contamination Control. This document was prepared by members of the Planetary Quarantine Department of Sandia Laboratories and will be published by the Government Printing Office in late 1967.

***

This work was conducted under Contract No. NASA-R-09-019-040, Bioscience Division, Office of Space Science Application, NASA Headquarters.

| Area | Problem |
|---|---|
| Medicine | - Limit (remove) virulent life forms, particulate matter, chemicals and gases on and in (from) instruments, drugs, environments and humans. |
| Foods | - Limit (remove) viable virulent life forms, harmful chemicals and gross particulate matter in (from) foods and environments. |
| Drugs | - Limit (remove) certain viable virulent life forms, harmful chemicals and particulate matter in (from) drugs and environments. |
| Manufacturing | - Limit (remove) certain types of gases, liquids and solids on and in (from) products, environments and raw materials. |
| Air Pollution | - Limit (remove) certain particulate material and chemicals in (from) air. |
| Water Pollution | - Limit (remove) certain particulate material, chemicals, life forms in (from) natural water sources. |
| Planetary Quarantine | - Limit (remove) viable micro-organisms on and in (from) space vehicles, environments, materials and parts. |

At least one important observation may be made about contamination control as represented by this list. It is this: a narrow view of contamination control techniques is not possible. It is not possible to view any technique as being exclusively associated with a given area because of the similarity of the problems arising in many areas. The recent use of laminar air flow clean rooms in medicine provides an excellent example of this.[2] Also, it is not possible to equate contamination control with any one technique because of the universality of the field. Cleaning, clean rooms, ultrasonication, and so forth, are all important in addressing the problems of contamination control.

Thus, for purposes of this paper, contamination control is viewed as a broad, important field in which the problems exhibit a similar abstract structure and the many techniques for their resolution may have wide applicability.

---

[2] McDade, J. J., Whitcomb, J. G., Rypka, E. W., Whitfield, W. J. and Franklin, C. M., The Microbial Profile of a Vertical Laminar Airflow Surgical Theater, Sandia Laboratories Research Report, SC-RR-67-456. This paper presents some specific results indicating the effectiveness of laminar airflow in actual surgical situations and, in addition, has a reasonable bibliography for persons interested in pursuing the subject further.

Planning for Contamination Control. There are several things which influence the author's belief that planning for contamination control is highly desirable. In this context, planning refers to (1) formulating objectives, (2) determining activities which will accomplish the objectives in some "optimal" fashion, and (3) deciding how the activities are "best" undertaken.

If only one means of resolving a problem is available, then planning consists primarily of (1) deciding whether to use it and control contamination or not to use it, with the opposite effect, and (2) making arrangements to use the one means available, if that is the decision. However, when many alternative means of resolving a problem are available, then it is often desirable to choose the "best" from among all alternatives. The view of contamination control expressed above, that is, a broad field of similarly structured problems, techniques for whose resolution may have wide applicability, inevitably leads to the conclusion that there may be many ways of resolving a given problem. With continued technical advances, this will become almost a certainty: leading to an increased need for selecting a "best" means of problem resolution from among the alternatives. Planning plays an important role in doing this.

The cost of contamination control activities in this country today must be enormous. For example, it has been estimated that as much as 275 billion dollars would be necessary over the next 34 years to resolve the air pollution problem.[3] If "best," in the preceding paragraph is related to "lease cost," one can begin to realize the potential importance of planning.

Not unrelated to the question of cost is the question of general "efficiency." Lower costs and, perhaps, greater results (faster, more effective) stem from efficient actions or activities to resolve problems. Both technical and administrative planning tend to encourage this efficiency.

Thus, planning seems desirable because (1) there is an almost inevitable increase in the number of ways of resolving problems (stemming from the broad view of contamination control) and (2) the potential savings and, possibly, technical gains, associated with the efficiency derived from planning seem great.

A theory of contamination control adequate for planning purposes should have several properties:

- a capability of addressing problems before they arise
- few limitations regarding the types of contamination or control techniques considered
- a capability to determine "most effective" means of achieving overall contamination control objectives.

A logical first step toward the development of a theory of contamination control is the formulation of a collection of general objectives

---

[3]The cost figure given here is the largest come across by the author. This figure and several others may be found in Hearings before the Subcommittee on Air and Water Pollution of the Committee on Public Works of the United States Senate, Ninetieth Congress. First Session on S 780, Part 2, p. 943.

to which all persons actively engaged in this activity can subscribe. Then some means of deriving "optimal" activities to achieve these objectives should be found. Accordingly, the remainder of this paper is devoted to a discussion of contamination control objectives, a means of relating these objectives to activities sufficient for the achievement of the objectives, and the notion of cost-effectiveness in contamination control.

It should be emphasized that some of the material that follows is subjective in character and represents only the current views of the author.

## The Systems Philosophy

In Theory. One philosophy which typically concerns itself with a broad, unified view of the subject being addressed is the "systems philosophy." Rather than appeal to other, often conflicting, definitions of systems analysis, systems studies, systems engineering, operations research, and so forth, a general outline of the "systems philosophy" (as seen by the author) is given below.[4]

A philosophy familiar to men for several centuries is that of the "scientific method." In general, one attempts to determine characteristics of "natural systems" by entering into a logical sequence of actions resembling those shown below.

THE SCIENTIFIC METHOD

| | |
|---|---|
| Natural System | Observe the System |
| | Model the System |
| | Verify the Model |
| | Analyze the Model |
| | Draw conclusions about the System |

A few comments about this list are in order. Observation of a natural system clearly depends upon a person's ability to observe. This ability to observe is not only a function of the state of technology and the system being observed, but also of the observer, himself.[5] Thus, subjectivity is inherent in observation. The phrase "Model the system" is often stated "Formulate Hypotheses".[6] "Model," in this context has a broad meaning: an abstract representation of the interrelationships

---

[4]Numerous similar descriptions of "operations research," "systems analysis" and so forth may be found in the literature. For example:
(i) Ackoff, R. L., "The Development of Operations Research as a Science," Operations Research, June 1956, 4:3, 265-287.
(ii) Hall, A. D., A Methodology for Systems Engineering, Van Nostrand, Princeton, 1962, pp. 19 and 140.
(iii) Optner, S. L., Systems Analysis for Business Management, Prentice-Hall, Englewood Cliffs, N. J., 1960, p. 31.

[5]Weyl, Hermann, Philosophy of Mathematics and Natural Science, Princeton University Press, 1949, Section 17.

[6]For example, (iii) of Footnote 4.

being observed.[7] The choice of a model is, again, a matter of judgment. It represents, in effect, the observer's "hypotheses" stated in some abstract form. The model is very much a function of the observations that are made. Verification of a model is rather difficult; science deals with a series of approximations to reality rather than with "truth." Hence, verification really means that there have been no reliable observations which are in contradiction with the model (insofar as the person doing the modeling knows), and there have been sufficiently many observations to lend credence to the belief that this will continue to be the case as long as one's ability to observe is unchanged. Thus, verification is not absolute. It depends upon personal and scientific judgment and upon one's ability to observe. Analysis of a model is normally an exercise in logic, but the completeness of an analysis can often be questioned. The conclusions are statements about the natural system that can be made as a result of the analysis of the model. As such, they are generally no more reliable than the observations, the model, and so forth.

In practice one may not follow the sequence Observe, Model, Verify, Analyze, Conclude in precisely that order. For example, if verification of the first model is impossible, the sequence may be Observe, Model, Observe, Model, Verify, and so forth. Similarly, if the conclusions are not consistent with the system being observed, the whole original sequence, or some portion of it must be repeated. Thus, the scientific method is a dynamic philosophy which leads to ever better approximations of "reality" based upon judgment and the ability to observe.

In "systems philosophy," as seen by the author, is very similar in character to the "scientific method." The major difference between the two is that the "natural system" of concern to one in the scientific method is replaced by a system over which one has some direct control: a "partially controllable system." Two comments should be made about this notion. First, control may be possible in at least two ways: physical and mental. Physical control refers to the ability to do things such as "control the humidity," "control the airborne particulate matter," and so forth. Mental control refers to the ability to make decisions such as "use a class 100 clean room," "Chemically clean the product," or to a creative sort of control (for example, the ability to design a product so that it is less likely to fail from certain types of contaminants). The second comment about a "partially controllable system" is that this phrase is really undefined. Somehow, the fact that "system," in this context, has yet to be satisfactorily defined causes some persons discomfort. Yet, "natural system" is, it would seem, equally undefined, and this appears to bother few people. This difference in attitude is probably due to the fact that numerous "natural systems" have been investigated by using the scientific method, and this has provided an intuitive base for thinking about "natural systems." On the other hand, it may be that many fewer "partially controllable systems" have been thoroughly analyzed using the "systems philosophy" described below, or it may simply be that such studies are not yet publicized adequately to provide this intuitive base for "partially controllable systems." In any event, it is hoped that the lack of a precise definition of a "partially controlled system" can be compensated for by the recognition of the similarity to the situation existing for a "natural system."

---

[7] For an enlightening discussion about models, see Bross, I.D.J., "Models," an article appearing in both: Design for Decision (by I.D.J. Bross), Macmillan, New York, 1953, pp. 161-182, and Scientific Decision Making in Business, (ed. Abe Shuckman) Holt, Rinehart, Winston, New York, 1963, pp. 63-77.

Having some control over a system normally implies that this control should be used to best advantage; that is, if one is dealing with a partially controllable system, he should have objectives stating what he desires as a result of his ability to control parts of the system. This is a fundamental concept in the systems philosophy, which may be outlined in the same form as the scientific method as follows.

SYSTEMS PHILOSOPHY

Partially Controllable System

- State Objectives (re control)
- Observe the System
- Model the System
- Verify the Model
- Analyze the Model
- Optimize
- Draw Conclusions (activities)

In outline, the systems philosophy differs from the scientific method in two areas. The first is a statement of objectives occasioned by the existence of some control over the system in question and the second is the possibility of drawing "optimal" conclusions from the modeling representing, in some sense, the "best" way to achieve the objectives. Fundamentally, the difference is deeper because of the various possible types of control and the latitude given in considering some concept or entity a "partially controllable system." Thus, for example, the system may be only an abstraction whose existence is implied by objectives which state its desired properties. Hence, in a real sense, the emphasis in the "systems philosophy" is upon the realization of objectives (dealing with control) through a sequence of actions similar to that occurring in the scientific method. While the conclusions to be drawn may deal with the uncontrollable aspects of the system to some extent, the basic conclusion is a collection of activities that, if implemented, will achieve the objectives "optimally."

The comments made about observation, modeling, and so forth, in the brief description of the scientific method above, apply to them as concepts in the systems philosophy. There are some additional comments appropriate here. Observation, in the application of the systems philosophy, may be an entirely intellectual affair if the system in question is only a concept. In this case, verification of any model is an intellectual activity either until the activities (conclusions) are implemented and the results compared with theory or until a reliable means of simulating the outcome a priori is developed. Sometimes the nature of the system precludes ever verifying the model (e.g., when the necessary testing would be prohibitively expensive). Optimization means essentially "select, from among all possible alternatives, one which best achieves the objectives." Of course, one must determine what "best" means, but even when it is possible to do this, there are several pitfalls. First, all possible alternatives are probably not known. If they are, an "optimum" may not exist because of the nature of the problem. Finally, if an "optimum" choice exists, it may not be possible to find it in practice because of theoretical or computational inabilities. Thus, the word "optimize," in practice, must be understood to imply an "attempt to optimize." The conclusions to be drawn are basically concerned with using the control one has. Thus they have been termed "activities." In essence, they represent a statement of what things must be done in order to "best" achieve the objectives.

The scientific method and the systems philosophy are very similar in intent. There is a distinction between the two in as far as one can distinguish between the investigation of interrelationships per se and the control of parameters appearing in such relationships. But this is often difficult, if not impossible, to do. For example, in investigating interrelationships, there is normally an objective: to do so in the "best" possible way. Thus, in investigating, one is in the position of applying the systems philosophy, which surely must have some effect upon the experimentation. Since activities stemming from both philosophies may well stem from the same model, differentiation between the two may be impossible except by a subjective evaluation of "intent." However, it should be emphasized that the intent in the systems philosophy is to determine activities which allow one to optimally achieve objectives.

In Practice. The systems philosophy, as outlined above, seems theoretically well-suited to planning: its intent is to answer the question "how should one act in order to optimally achieve his objectives?" But there is another "how" that seems quite apparent. How does one apply the systems philosophy? The final answer, of course, must depend upon circumstances, but some things may be said about this "how." The preceding material was not new - only the wording, and perhaps emphasis, has been altered to conform to the subject of the paper. However, what follows is fairly original, and subject to considerably more scrutiny!

In approaching a system with the systems philosophy, one has given a system and objectives. The objectives need not be precise, but should convey intent about the desired results of control of the system. The approach outlined here will be to operate primarily from the objectives with the system providing constraints upon actions. Before proceeding, a few terms will be defined.

For purposes here, an objective is considered to be a statement which contains or implies the existence of variable factors and which specifies some desirable behavior or value for the variable factors. So, for example, the objective "to cut monthly costs in the future" contains at least two variable factors: cost and time. The objective postulates that these are related, and that at some future time, costs should be lower than at the time the objective was stated. Had the objective been stated "to cut monthly costs $100,000 beginning next month" the existence of the cost and time variables is implied, and this objective specifies values for the variables. In effect then, the word objective will be considered synonomous with a statement indicating desired behavior or values for variable factors which the objective formulator wishes to have controlled, influenced or measured.

It should be remarked that not all statements commonly thought of as objectives completely satisfy this definition. The basic reason for this is the existence of social, environmental, technological, and other norms which make it unnecessary to state the variable factors and specify their desired behavior. For example, the statement "to determine the length of a given room" might be of this type. If a man is already in the room with a tape measure, there is an implied desire for a reasonably immediate answer, for accuracy of measurement compatible with that obtained from a tape measure, and for a cost commensurate with this type of activity.

In attaining an objective, the person responsible for its attainment frequently pays a penalty. This penalty may be in the form of a

dollar cost or any other expenditure of resources (time, personnel, and so forth). Sometimes the penalty is a loss of other desired goals. When an objective involves a desire for improved efficiency, it may be the case that no penalty is incurred by the person responsible for the attainment of the objective. However, in this case, it may be that other persons, contractors for example, sustain a loss, or penalty, so that the notion of a penalty can depend highly upon one's point of view.

In general, there is a spectrum of types of objectives that may be stated either by an individual or a group acting in unison. These range from free objectives to bound objectives.

Free objectives are usually conceptual in nature and recognize a need for some general outcome or type of behavior without specifying the "amounts." For example, the objective "cut monthly costs in the future" is of this type. The general intent of such a statement is clear, even though one may meet this objective by cutting monthly costs by any amount at any future time. Presumably it is left to the person or persons responsible for achieving the objective to determine what reasonable cost cuts are in any given time period.

A bound objective, on the other hand, is one that is specific in nature. For example, "cut monthly costs $100,000 starting next month" is bound since it requires few if any decisions about the objective itself by those responsible for achieving it.

There are objectives which lie somewhere between these two extremes. These have some elements of constraint and some elements of choice for the implementer. An example of such an objective might be "cut monthly costs beginning next month."

Free objectives, as envisioned here, contain a maximum amount of variable quantities. As such, free objectives may be regarded as abstract statements of intent or desire. The same free objective may assume many bound forms depending upon the values or specific behavior desired for each of the variables. For example, "to cut monthly costs $100,000 beginning next month" and "to cut monthly costs $25,000 beginning in three months" are two bound objectives derived from the free objective "to cut monthly costs in the future."

It is assumed that, whenever the systems philosophy is employed to determine what activities are needed to "best" achieve objectives relating to a partially controllable system, ultimately some objective or objectives must be bound. For example, when a free objective "cut monthly costs in the future" is stated the ultimate determination of activities to accomplish this objective involves either an a priori or a posteriori statement of the specific amounts (to be) saved in any given month, and this latter statement may be regarded as a bound objective. Because of our interest in planning, it is assumed that an a priori bound objective is needed. This is the antithesis of taking some action and then assessing its effectiveness.

Accordingly, the emphasis in the following material is on two items: a possible means of relating objectives to actions which will achieve them with an acceptable penalty, and doing so in such a fashion that a bound objective with an acceptable penalty may be stated before the advent of any action designed to meet the associated free objective.

The objectives associated with the systems philosophy are called primary objectives. These objectives provide the raison d'etre for the

activities undertaken to control the system and provide also criteria against which the success of the activities may be judged. Normally, in any large program, one would not expect these objectives to be directly achievable, that is, the means for directly controlling the variables occurring in the objectives so that the objectives may be achieved, are not known. When this is the case, the variables occurring in the objective must be analyzed to determine the activities necessary for the attainment of the objective.

The analysis of primary objectives leads to a consideration of the "significant factors" which influence their attainment. For example, it is not unreasonable to imagine that the factors "salaries" and "purchases" influence the monthly cost incurred by an organization. The determination of a set of all such "significant factors" is often a matter of judgment. The relationships between the primary objectives and the "significant factors" influencing their attainment is, similarly, a matter of judgment, and are expressed in the form of a model (or models), with due consideration being given the system being modeled.

The desire to attain primary objectives implies the existence of certain objectives relating to the significant factors associated with the primary objectives. For example, if "salaries" and "purchases" are deemed to be the only significant cost factors of an organization, then a model relating these to organizational cost might take the simple form

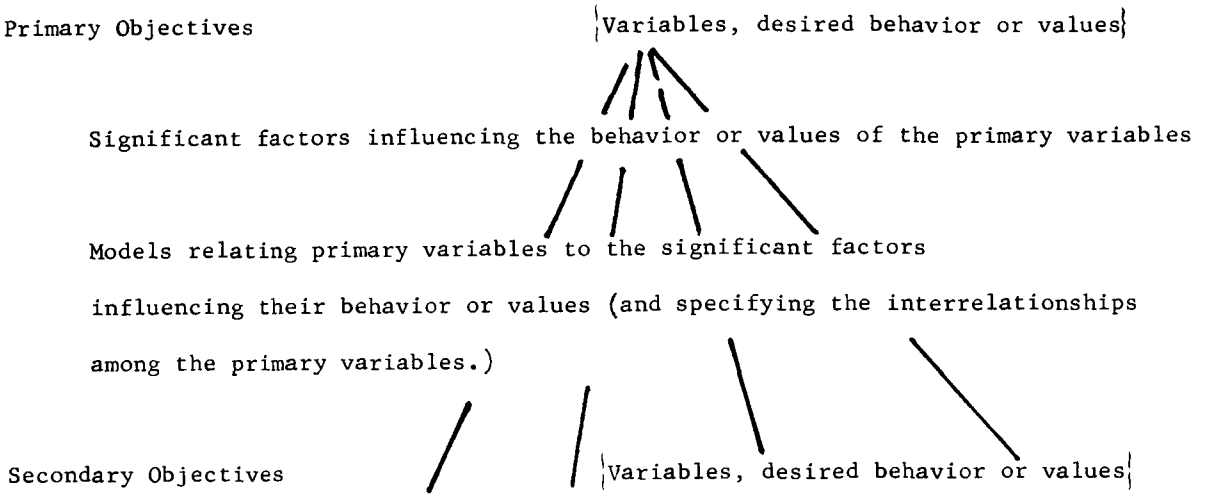
$$C = S + P$$

where C represents total organizational dollar cost per month, S represents total organizational salary cost per month and P represents the total purchase cost per month. Each of the quantities C, S and P may vary with time, or

$$C(t) = S(t) + P(t).$$

Then, for example, the objective "cut monthly costs in the future" may be interpreted as specifying that at some time $T > 0$, $C(t)$ should be less than $C(0)$ for $t \geq T$. This, of course, is only one possible interpretation. In this example, $S(t)$ and $P(t)$ represent the factors which influence the achievement of the objective. Depending upon the objective implementer's decision, the original objective, $C(t) < C(0)$ for $t \geq T$, implies either that $S(t) < S(0)$ or that $P(t) < P(0)$ for $t \geq T$ (or both). Thus, the original primary objective implies the existence of objectives dealing with the significant factors influencing the achievement of the primary objective.

The new objectives associated with the significant factors influencing the achievement of the primary objectives are called <u>secondary objectives</u>. In essence, these secondary objectives are statements about the desired mode of behavior or value of the variables representing the significant factors influencing the achievement of the primary objective. The word variables or parameters is appropriate here as long as the significant factors occur in some parametric form. This notion is outlined schematically on the next page.




Secondary objectives are highly dependent upon the nature of the primary objective, the choice of significant factors influencing the behavior or possible values of the primary variables and the choice of a model to relate these. The hope is that if the secondary objectives are achieved, the primary objectives will be also. This type of behavior is evident in the simple model used for illustrative purposes above; that is, if

$$\text{(i)} \quad S(t) - S(0) \leq a,$$

$$\text{(ii)} \quad P(t) - P(0) \leq b$$

and

$$\text{(iii)} \quad a + b < 0 \text{ for } t \geq T,$$

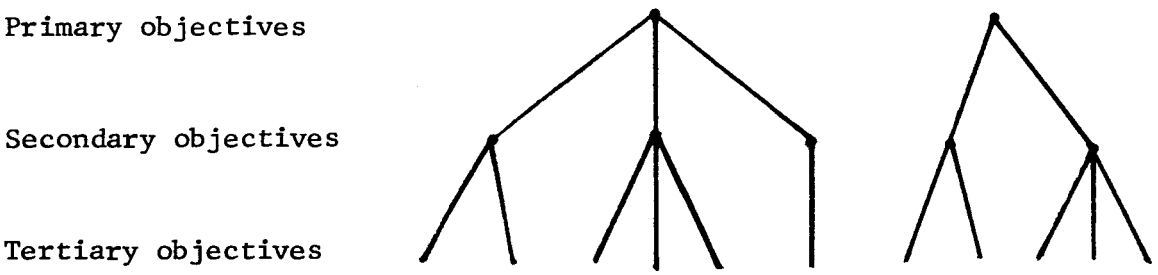
then one must have

$$C(t) - C(0) < 0 \text{ for } t \geq T$$

If S(t) and P(t) are the only significant factors influencing C(t), and if the simple model represents the relationship in existence between these, then the attainment of all of the three secondary objectives given above implies the attainment of the primary objective "to cut monthly costs in the future."

A similar analysis of the secondary objectives may then be undertaken. This analysis yields a collection of tertiary objectives which play the same role to the secondary objectives as the secondary objectives do to primary objectives. This process may be continued to yield 4th level objectives, 5th level objectives, and so forth.

One feature of a process such as this is that it has a tree-like or branching structure. That is, a single primary objective may yield several secondary objectives each of which, in turn, may yield several tertiary objectives, and so forth. Thus the structure is similar to that shown on the next page.




Clearly there is no advantage to a scheme such as that outlined above unless it aids one in determining what activities can be undertaken to achieve the primary objectives with an acceptable penalty.

In order to see how it might do this, the variables associated with any objective are divided into two classes: those variables which are actionable and those which are not. An actionable variable is one which can be directly controlled or measured. As discussed earlier, control may occur in one of two ways and is a fairly subjective matter. Physical action may be taken to control the variables in a predictable fashion, or the variables may be controlled by fiat (e.g., a decision about the magnitude of the variable). There may be variables over which one has no control, and these are actionable if they can be directly measured with an acceptable degree of accuracy. An example of an actionable objective is the objective

$$a + b < 0 \text{ for } t \geq T,$$

above, since this may be controlled by the person performing the analysis.

The intent in constructing a tree-like hierarchy of program objectives is, then, that each branch of the tree should ultimately terminate with an objective each of whose variables is actionable. If this can be done, then one has a scheme which relates the primary objectives of the program to activities that must be taken in order to achieve these primary objectives. Such a statement must be tempered by the realization that its validity depends upon the completeness of the sets of "significant factors" and the appropriateness of the choices of the models occurring throughout the structure.

So far, only the relating of objectives to actions designed to achieve them has been considered. No attention has been given to the possibility that the necessary actions will involve too great a penalty. Formation of a tree-like hierarchy of objectives, each branch of which terminates in objectives containing only actionable variables, may be accomplished independent of the location of the primary objectives on the free-bound scale. The simple illustration "to cut monthly costs in the future" is an example involving a free primary objective.

When one begins with free primary objectives, then all other objectives in the hierarchy are free also. In particular, terminal objectives involve actionable variables whose desired modes of behavior or values are specified abstractly, as for example in the simple model presented earlier where $S(t) < S(0)$ or $P(t) < P(0)$. In this simple example, to have a bound primary objective, one must specify how much less $C(t)$ should be than $C(0)$ when $t \geq T$, and T must also be specified. This can certainly be done directly, or it can be done by specifying T and the

relationships between S(t) and S(0) and between P(t) and P(0). If, for example we let

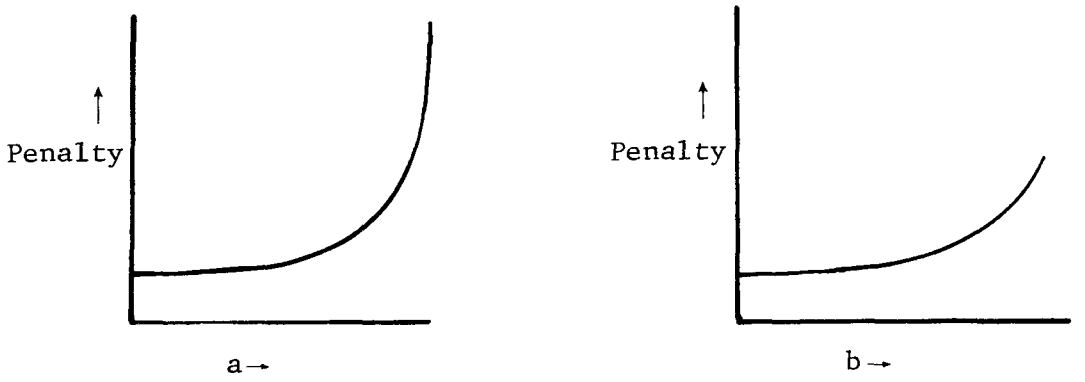
$$S(0) - S(t) = a$$

and

$$P(0) - P(t) = b$$

and if there are penalties known for values of a and b as shown below, then an analysis may be performed to obtain the optimum a and b for a given requirement on

$$a + b = C(0) - C(t).$$




There may be a different set of such curves for each specific value of T. Thus for each bound primary objective, that is, specification of C(0) - C(t) = a + b and T, one can determine the minimum penalty that must be incurred to achieve the specific values. If there is then a utility associated with each possible combination of C(0) - C(t) and T, it is possible to choose some combination for which the utility per unit penalty is maximal. In the absence of a well-defined utility measure, it is still possible to choose at least one combination of C(0) - C(t) and T which has an acceptable penalty associated with it. Thus, the objective hierarchy may be useful both in relating objectives to activities designed to achieve them and also in determining bound program objectives which have acceptable associated penalties. When utilities for specific primary objectives are known, an "optimal" solution may be obtainable.

In general, the objective hierarchy terminates in a collection of objectives each of which may be directly achieved (i.e., they possess only actionable variables). These are normally free objectives in that they are stated in some abstract parametric form, as the example above illustrates.

For any assignment of specific parameter values in all of these terminal objectives, one can determine the resources needed to achieve them. Notationally, if $\beta_1,\ldots,\beta_M$ represent the variables occurring in the terminal objectives, in theory one can obtain an approximate penalty function

$$P(\beta_1,\ldots,\beta_M)$$

by (1) determining the "best way of achieving the terminal objectives as a function of the values of the variables occurring in them,
(2) listing the resources needed for the attainment of the terminal objectives individually (as a function of the parameters),
(3) eliminating "redundancy" in resources (for example, if a certain facility may be used to control two variables, possibly occurring in different terminal objectives, it is included in total resources only once)
(4) translating total resource expenditure into penalty units.

Then, for bound (specific) primary objectives, one is attempting to determine specific values for the parameters $\beta_1,\ldots,\beta_M$ so that the primary objectives are achieved and $P(\beta_1,\ldots,\beta_M)$ is minimal. This essentially determines the "optimal" activities needed for the achievement of any bound primary objectives.

Finally, to aid in selecting acceptable bound primary objectives, one may vary the specific values appearing in them and determine the penalty associated with "optimal" attainment of each bound objective so obtained. If utilities for bound primary objectives are known, then a utility-penalty analysis may be performed to determine "best" primary objectives. If utilities are not known, one still may seek a bound primary objective having an "acceptable" penalty associated with its achievement.

The approach to utilization of the systems philosophy outlined above gives one a framework with which to answer the question "how does one implement the systems philosophy?" This is done by changing the problem to one of finding significant factors and models to subproblems. In practice, this seems to provide more order to the use of the systems philosophy. The "model" of the systems philosophy becomes, in this context, a collection of models associated with the objective hierarchy. Optimization takes the form of a utility-penalty analysis of the hierarchy, and the conclusions (activities) are determined by the actionable variables. The "partially controllable system" appears throughout the hierarchy as a constraint upon the "significant factors" and the models that are chosen.

## A Systems Approach to Contamination Control

General Comments About an Objective Hierarchy. Early in the paper, it was pointed out that contamination control was generally concerned with limiting or removing solid matter, gases or liquids in, on or from other solids, gases and liquids. But, is this a sufficient description of contamination control planning objectives? The answer is, probably not; since this statement really yields no way of determining the amount of control needed - an essential for planning.

The fact is that contamination control is a field which serves higher objectives, and its role is best understood by considering these objectives. If there were no penalty associated with the existence of contamination in a given situation then there would be no reason for contamination control. The following list gives some indication of the types of penalties encountered in areas where contamination control is practiced.

| Area | Penalties |
|---|---|
| Medicine | - loss of health |
| | - death |
| | - needless suffering |
| Foods | - loss of health |
| | - death |
| | - unfavorable FDA action |
| | - loss of business |
| Drugs | - loss of health |
| | - death |
| | - unfavorable FDA action |
| | - loss of business |
| Manufacturing | - product failure |
| | - unnecessary expense |
| | - loss of health |
| | - unfavorable government action |
| | - loss of business |
| Air Pollution | - loss of health |
| | - human inconvenience |
| | - loss of native flora |
| | - esthetic loss |
| Water Pollution | - loss of health |
| | - death |
| | - loss of native biota |
| | - esthetic loss |
| | - loss of recreational areas |
| Planetary Quarantine | - loss of scientific information about planets |

Thus, contamination control activities are desired in each area because of the penalty associated with the lack of them. But more than this, in any given situation the penalty that one pays depends upon the amounts of contamination of various kinds that are present. For example, in many medical situations the "normal" environmental infectious contamination for most micro-organisms is acceptable without measurable penalty, whereas in others (severe burns, transplant patients) infectious contamination should be as low as possible because of the severe penalty if the situation is otherwise.

Hence, not only do penalties imply the possible need for control but they also give some insight into "acceptable" levels of contamination. This is important for planning activities in contamination control since the control technique chosen must depend upon the amount of contamination that is permissible, and the cost incurred in contamination

control will be a function of the control techniques chosen. Thus, to plan for contamination control, one must have some knowledge of "acceptable" levels of contamination. It might be expected that knowledge of "acceptable" levels of contamination will come from persons outside of the contamination control area. For example, it would probably take a medical specialist to determine "acceptable" levels of types of air contaminants when the penalty for their existence is primarily medical in character. Nevertheless, contamination control is highly dependent upon the existence of penalties incurred in its absence.

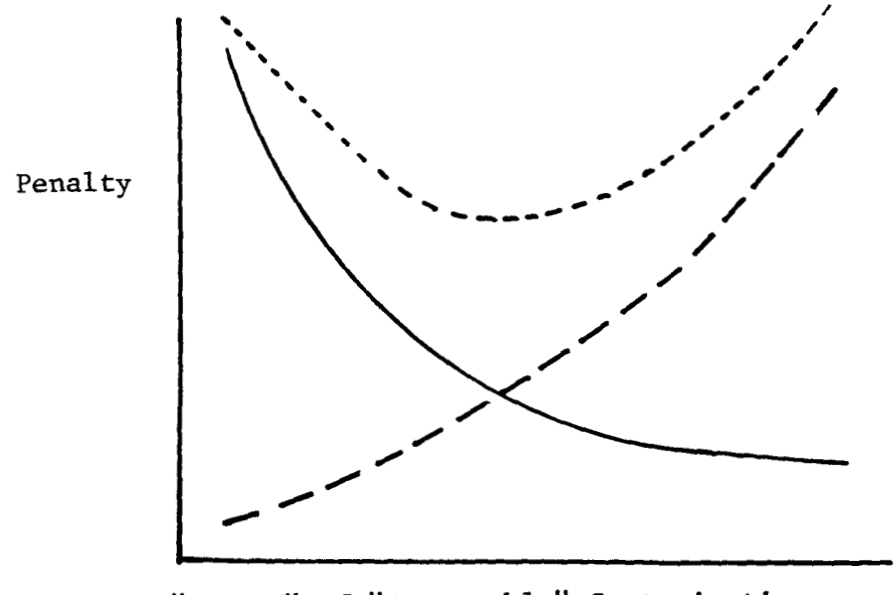
With this in mind, one goal of contamination control activities is:

Goal 1. To control contamination so that the payment of unacceptable penalties due to contamination is avoided.

There are other goals also. For example, there is usually a penalty associated with the control of contamination as well as with its existence since control normally requires an expenditure of resources. Hence, in planning for contamination control, one wishes also:

Goal 2. To achieve Goal 1 so that the penalty associated with the control activities is acceptable.

Not surprisingly, planning must also concern itself with the total acceptability of both of these penalties. If both the penalty for the existence of contamination and the penalty for controlling contamination can be expressed in common units (dollars, for example) then the situation might resemble that shown in the figure below. In this figure, it is assumed that the "amount" of contamination that is acceptable depends not only upon the penalty one pays due to its existence, but also upon the penalty one pays for its control. Thus the "acceptable amount" may be treated as a variable until both these things are known as a function of "acceptable amount."



In this simple illustration, the total acceptable penalty, which, incidentally, is a decision in this setting, must be compared with the curve representing the sum. If that curve is always above (greater than) the total acceptable level, then the problem cannot be resolved without altering the notion of acceptability or finding means of control with a lower associated penalty. Thus, while it is appropriate to consider the penalty from the existence of contamination, and its acceptability, per se, these will often be influenced by the penalty paid for control. For example, if the maximum acceptable probability of failure of a device from a contamination failure mode is initially assumed to be $10^{-3}$, but it is later found that the cost of attaining this level (by controlling contamination) is ten times that associated with $3 \times 10^{-3}$, one might wish to reconsider his original notion of acceptability. Generally speaking, the notion of acceptable penalties associated with the existence of contamination involves some elements of judgment.

Often, curves like those in the above figure are not available: either because the penalty units are different for the existence and control of contamination or because the penalty associated with the existence of contamination is not thoroughly understood. The latter might be the case, for example, in planetary quarantine. In any event, the penalty paid for control should be considered in planning for contamination control.

Before attempting to formulate general primary objectives for contamination control, it is convenient to note that the actual achievement of Goal 1 is often difficult to determine. This is so for at least two reasons. First, it is generally not possible to monitor or measure amounts of contamination exactly because of the inadequacy of measurement techniques, the complexity of the total system (including control environments, nature of contamination) and so forth. Secondly, in many contamination control situations it is not possible to obtain sufficient data about the adequacy of the contamination control undertaken, in terms of results, to know with certainty that the goal has been achieved. Planetary quarantine may, again, be such a situation. Thus, it may be more appropriate to speak of the probability of achieving Goal 1, and this goal may be rephrased:

OBJECTIVE 1: To control contamination so that the probability

$$\Pr\{\text{payment of unacceptable penalties from contamination}\} \leq \epsilon .$$

This form has certain advantages in that it:

- recognizes the possible uncertainty in knowledge about the achievement of Goal 1, and
- allows for trade-offs to be made between $\epsilon$ and the penalty for controlling contamination.

Combining Objective 1 with Goal 2 leads to the objective statement

A CONTAMINATION CONTROL OBJECTIVE. To control contamination so that

$$\Pr\{\text{payment of unacceptable penalties from contamination}\} \leq \epsilon$$

without incurring unacceptable penalties as a result of the control activities.

The nature of the control to be exercised, the control activities, are unknowns in this objective, and the object is, in essence, to determine them. Hence, "control" and "control activities" represent variables in the objective. There are other variables as well. Because of the interdependence of the "acceptable penalties" of both types, it is undesirable to make these specific until the interrelationship is understood. Thus, "acceptable penalties" in both usages in the objective is a variable. Also, the parameter $\epsilon$ is a variable since, generally, the penalty incurred from control depends upon the value of $\epsilon$, and it is desirable to understand the functional relationship between the two to aid in deciding what the ultimate value of $\epsilon$ should be. Certainly there will be other information available to aid in this also. Its possible that "contamination," itself, is a variable in the sense of the preceding section. Finally, the probability

$$\Pr\{\text{payment of unacceptable penalties from contamination}\}$$

must be regarded as a variable because one presumably has some control over its value.

In constructing an objective hierarchy from the Contamination Control Objective just stated, it is convenient to note that the second part of the objective, corresponding to Goal 2, need not be included in the primary objective of the hierarchy. This occurs because a hierarchy constructed from Objective 1, yields a means of attaining the second part of the Contamination Control Objective as outlined in the previous section. Thus, it is necessary only to construct the objective hierarchy from Objective 1. If $P_{un}$ is used to designate

$$\Pr\{\text{payment of unacceptable penalties from contamination}\},$$

the primary objective to be analyzed takes the form:

$$P_{un} \leq \epsilon \ .$$

As we begin to construct an objective hierarchy from this primary objective, it is worth reiterating that

- the choices of models and significant factors are subjective matters, and
- in actual usage, these choices would be related to the specifics of the situation.

Thus, only a few "levels" of the tree will be constructed as a means of illustrating the relationship between contamination control techniques and activities to general objectives.

What might the significant factors influencing the behavior of $P_{un}$ be? Some factors which must ultimately influence its behavior are

- The ways in which unacceptable penalties may be incurred from contamination,
- Types and amounts of contamination involved in these ways of incurring unacceptable penalties, and
- Sources of the types of contamination being considered.

Suppose there are N independent ways of incurring unacceptable penalties from contamination, and that an unacceptable penalty is incurred over-all if an unacceptable penalty is incurred in any of these N possible ways. "Independent" means, roughly, that incurring an unacceptable penalty in one way does not influence the probability of incurring an unacceptable penalty in any other way. Let

$P_{un}^{(i)}$ - represent the probability of payment of unacceptable penalties from contaminating the $i^{th}$ way.

Here, $i = 1, 2, \ldots, N$. A simple example of this situation might be a system containing two valves in which the system fails whenever either valve fails and in which the failure of one valve does not influence the behavior of the other valve.

Then the $P_{un}^{(i)}$, $i = 1, 2, \ldots, N$, may be regarded as a set of "significant factors" influencing $P_{un}$. Under the conditions imagined, the relationship between the $P_{un}^{(i)}$ and $P_{un}$ may be expressed

$$P_{un} = 1 - \prod_{i=1}^{N} \left[1-P_{un}^{(i)}\right].$$

Since $P_{un}$ is desired to be no greater than $\epsilon$,

$$1 - \prod_{i=1}^{N} \left[1-P_{un}^{(i)}\right] \leq \epsilon$$

or

$$\prod_{i=1}^{N} \left[1 - P_{un}^{(i)}\right] \geq 1 - \epsilon .$$

This requires, in effect, that for $i = 1, 2, \ldots, N$,

$$1 - P_{un}^{(i)} \geq 1 - \epsilon_i$$

where

$$\prod_{i=1}^{N} \left(1-\epsilon_i\right) \geq 1 - \epsilon .$$

Thus, there are N + 1 secondary objectives

$$P_{un}^{(i)} \leq \epsilon_i, \quad i = 1, 2, \ldots, N$$

and

$$\prod_{i=1}^{N} \left(1-\epsilon_i\right) \geq 1-\epsilon.$$

If these are satisfied, then $P_{un} \leq \epsilon$, as desired.

Hence, the first level of the objective hierarchy may be represented schematically as

Primary Objectives: $P_{un} \leq \epsilon$

Model: $$P_{un} = 1 - \prod_{i=1}^{N} \left[1 - P_{un}^{(i)}\right]$$

$$\left(\text{Significant Factors } P_{un}^{(i)}\right)$$

Secondary Objectives: $P_{un}^{(i)} \leq \epsilon_i, \quad \prod_{i=1}^{M} (1-\epsilon_i) \geq 1-\epsilon.$

The secondary objective $\prod_{i=1}^{N} \left(1-\epsilon_i\right) \geq 1-\epsilon$

contains only actionable variables in the sense that one can presumably force this to be the case in his analysis. Thus this objective need undergo no further analysis. Hence, let us proceed to analyze the secondary objective

$$P_{un}^{(i)} \leq \epsilon_i.$$

Let us suppose that there are $M_i$ types of contamination which will contribute to the payment of unacceptable penalties in the $i^{th}$ way. Then, suppose that

$P_{un}^{(i)} \; a_1, a_2, \ldots, a_{M_i}$

- the probability of incurring unacceptable penalties in the $i^{th}$ way when there is an amount $a_j$ of the $j^{th}$ type of contamination ($j = 1, \ldots, M_i$) <u>available</u> for the $i^{th}$ way of incurring penalties,

and

$P_i \left( a_1, a_2, \ldots, a_{M_i} \right)$

- the probability that an amount $a_j$ of the $j^{th}$ type of contamination ($J=1, \ldots, M_i$) <u>is</u> available for the $i^{th}$ way of incurring penalties

are known. If the possible amounts, $a_j$ are discrete valued and lie in ranges

$$0 \leq a_j \leq \alpha_j$$

then

$$P_{un}^{(i)} = \sum_{a_1=0}^{\alpha_1} \sum_{a_2=0}^{\alpha_1} \cdots \sum_{a_{M_i}=0}^{\alpha_{M_i}} P_{un}^{(i)} \left(a_1, a_2, \ldots, a_{M_i}\right) \left(P_i \; a_1, a_2, \ldots, a_{M_i}\right)$$

Hence, one may consider the 2N probabilities

$$P_{un}^{(i)}(a_1,\ldots,a_{M_i}),\ i = 1,\ldots,\ N$$

and

$$P_i\ (a_1,\ldots,a_{M_i}),\ i = 1,\ldots,\ N$$

as significant factors influencing the attainment of the secondary objectives

$$P_{un}^{(i)} \leq \epsilon_i,\ i = 1,2,\ldots,\ N.$$

The secondary objectives, just stated, imply conditions on the behavior of the probabilities

$$P_{un}^{(i)}(a_1,\ldots,a_{M_i}) \text{ and}$$

$$P_i\ (a_1,\ldots,a_{M_i})$$

as functions of the "amounts" $a_1,a_2,\ldots,a_{Mi}$. These conditions become the tertiary objectives, and usually their exact form will be influenced by the specific nature of the system in question. No attempt will be made to derive them here in any general form. However, the tertiary objectives will involve specifying behavior for these 2N probabilities, so that analysis of them is appropriate.

In order to determine the significant factors influencing the behavior of $P_i(a_1,\ldots,a_{M_i})$, the probability that an amount $a_j$ of the $j^{th}$ type of contamination ($j = 1,\ldots,M_i$) is available for the $i^{th}$ way of incurring penalties, it seems reasonable to consider <u>sources</u> of contamination. Suppose there are K sources of contamination in the system, each of which may supply any or all pertinent types of contamination. Suppose further that they are independent sources in the sense that the effect of any one source upon the $i^{th}$ way of incurring penalties in no way influences the effect of any other source. Then, if from the $k^{th}$ source one gets an amount $b_{jk}$ of the $j^{th}$ type of contamination, this determines an array

$$\begin{matrix} b_{11}, & b_{21}, \ldots, & b_{M_i 1} \\ b_{12}, & b_{22}, \ldots, & b_{M_i 2} \\ \vdots & \vdots & \vdots \\ b_{1K}, & b_{2K}, \ldots, & b_{M_i K}. \end{matrix}$$

Suppose one has knowledge of the probabilities

$$P_{ik}\left(b_{1k},\ldots,\ b_{M_i k}\right)$$

- the probability that amounts, $b_{jk}$, of the $j^{th}$ type of contamination from the $k^{th}$ source are available for the $i^{th}$ way of incurring penalties,

(where $k = 1,\ldots,K$) for all possible arrays of the type above for which

$$\sum_{k=1}^{K} b_{jk} = a_j,\ j = 1,\ldots,\ M_i.$$

Then

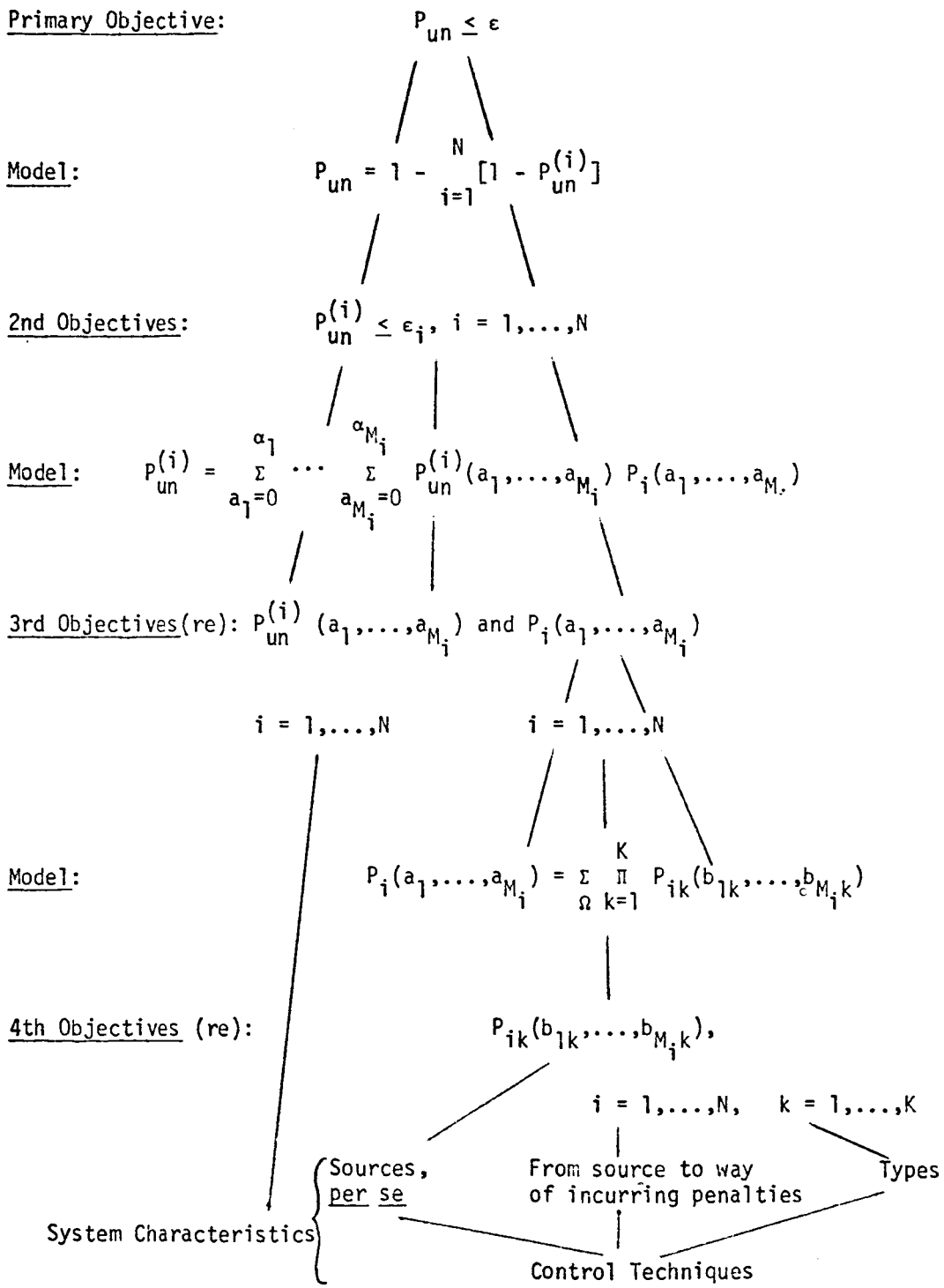
$$P_i\ \left(a_1,\ldots,a_{M_i}\right) = \sum_{\Omega} \prod_{k=1}^{K} P_{ik}\ \left(b_{1k},\ldots,b_{M_i k}\right)$$

where the summation extends over the set $\Omega$ of all K-by-$M_i$ arrays $\left|\left|b_{jk}\right|\right|$ for which

$$\sum_{k=1}^{K} b_{jk} = a_j,\ j = 1,\ldots,\ M_i.$$

At this point, the objective hierarchy is adequately developed to allow some insight into the relationships existing between general objectives and contamination control activities. While these relationships are to be viewed as a consequence of a number of assumptions (N,$M_i$ and K: known, ways of incurring penalties, sources: independent) it is very likely the case that a similar hierarchy exists when the assumptions are not valid. In this case, different models would be needed, but the nature of the parameters would probably not undergo "significant" change.

Referring to the objective hierarchy reproduced on the next page, it may be viewed intuitively as follows. The primary objective is of a general nature: control contamination to keep the likelihood of incurring unacceptable penalties arising from its existence small (less than "$\epsilon$"). It was then postulated that unacceptable penalties could be incurred in several ways, and the secondary objectives are statements implying a desire to maintain the likelihood of incurring unacceptable penalties in any way small (the $i^{th}$ way less than $\epsilon_i$). The "smallness," $\epsilon_i$, in this case, is directly related to the original quantity $\epsilon$. At the third level, it was postulated that the penalty incurred in any of the possible ways depended upon the type and amount of contamination available (for each way). Accordingly, the third level objectives become statements about (a) the likelihood of incurring unacceptable penalties in each way from defined types and amounts of contamination, and (b) the likelihood of actually having certain amounts of various types of contamination available for the $i^{th}$ way of incurring unacceptable penalties. These objectives were not explicitly stated because the nature of the system will influence their exact form. Finally, at the fourth level, the existence or availability of certain amounts of various types of contamination was postulated to depend upon the sources


Primary Objective: $P_{un} \leq \varepsilon$
Model: $P_{un} = 1 - \prod_{i=1}^{N} [1 - P_{un}^{(i)}]$
2nd Objectives: $P_{un}^{(i)} \leq \varepsilon_i$, $i = 1,\ldots,N$
Model: $P_{un}^{(i)} = \sum_{a_1=0}^{\alpha_1} \cdots \sum_{a_{M_i}=0}^{\alpha_{M_i}} P_{un}^{(i)}(a_1,\ldots,a_{M_i})\, P_i(a_1,\ldots,a_{M_i})$
3rd Objectives(re): $P_{un}^{(i)}(a_1,\ldots,a_{M_i})$ and $P_i(a_1,\ldots,a_{M_i})$
$i = 1,\ldots,N$
$i = 1,\ldots,N$
Model: $P_i(a_1,\ldots,a_{M_i}) = \sum_{\Omega} \prod_{k=1}^{K} P_{ik}(b_{1k},\ldots,b_{c_{M_i}k})$
4th Objectives (re): $P_{ik}(b_{1k},\ldots,b_{M_ik})$,
$i = 1,\ldots,N$, $k = 1,\ldots,K$
Sources, per se
From source to way of incurring penalties
Types
System Characteristics
Control Techniques

of contamination. As a result, the fourth level objectives become statements about the likelihood of certain amounts of contamination of various types being (i) available at each source, and (ii) capable of "reaching" the various ways of incurring unacceptable penalties. At each level, the postulates just mentioned represented the assumption of the "significant factors" appearing in Section II. In each case, mathematical models were used to relate these significant factors to the preceding variables. At one point, an actionable objective (containing only actionable variables) was found, and, in all probability, there would have been others had the third and fourth level objectives been stated precisely. In general, the objective hierarchy is compatible with the theory outlined in the section on "The Systems Philosophy."

Let us examine the consequences of developing the hierarchy this far. Down the right branches of the hierarchy (through "sources"), one's only concern is with sources of various types of contamination, and the availability of contamination from these sources for the various ways of incurring unacceptable penalties. Objectives associated with these items (at, in fact, the fifth level) are attainable in one or both of two broad ways. The first is physical control of contamination. This may be accomplished at its source or somewhere "between" the sources and the ways of incurring unacceptable penalties. The second is a "design" control to be discussed later. The fifth level objectives, in any specific problem, would pertain to the "allowable" amounts (parametrically) from any source or between any source and any way of incurring unacceptable penalties. Thus, hopefully, one could determine the "best" means of control as a function of the original $\epsilon$. Then $\epsilon$ could be determined as a function of the penalty paid for control.

This "optimization" cannot be accomplished, however, without consideration of the left branches of the tree. In this branch, one is concerned with the likelihood of incurring unacceptable penalties from each way of doing so when there is a certain amount of each pertinent type of contamination present. In any specific problem, one would hope to be able to determine this through further analysis coupled with experimentation. But there is a potential contamination control problem here that is often overlcoked. If the system is not completely designed, the possibility of control in these branches exists. That is, one may be able to design the system so that the likelihood of incurring unacceptable penalties from various types of contamination is small even though the amount of many or all types of contamination is large.

Similarly, the number and nature of sources, number and nature of the ways in which unacceptable penalties may be incurred, and types and modes of transport of contamination available may possibly be controlled through design. Thus, as might be intuitively obvious, system characteristics influence the complete tree. When these may be controlled through decisions, these decisions should be made so that the final physical control of contamination has a small penalty associated with it (insofar as possible).

To reiterate, using an approach such as this, it should be possible, at least in theory, to

- determine "optimal" means (activities, equipment, etc.) needed to achieve the contamination control objective for a given $\epsilon$ (allowable uncertainty in incurring unacceptable penalties),
- allow one insight into the dependence of $\epsilon$, and, indeed, the definition of an "unacceptable penalty from contamination," upon the penalty paid for controlling contamination,

- help one assess the effect of system design upon contamination control problems,
- aid in recognizing areas in need of investigation, and
- provide a common framework in which many, if not all, contamination control problems may be viewed.

Two Partial Examples. To illustrate the above, somewhat abstract, approach to resolving contamination control problems, two somewhat more specific problems in which this approach has been partially implemented will be briefly discussed.

Tierney and the author have considered the probability of failure from contamination of a valve.[8] In this instance, the primary objective might be stated: To control contamination so that the probability of failure of a valve before time T from particulate contamination should be less than $\epsilon$. In this primary objective, an "unacceptable penalty" appears in the form "failure before time T." In the situation envisioned, an unacceptable penalty was incurred in only one way: failure of one valve before time t. Thus the secondary objective was merely a rephrasing of the primary objective, i.e.,

$$P_F(t) \leq \epsilon \text{ for } t < T,$$

where $P_F(t)$ represents the probability of failure (from particulate contamination) at time t.

In the next level, it was hypothesized that failure occurred as a result of having certain amounts $a_i$ of M types of particulate contamination (here, $i = 1,2,\ldots,M$), and the analysis was much the same as the general case above with $\alpha_i = \infty$. That is

$$P_F(t) = \sum_{a_1=0}^{\infty} \sum_{a_2=0}^{\infty} \cdots \sum_{a_M=0}^{\infty} P_F(a_1,\ldots,a_M:t)\, P(a_1,\ldots,a_M:t)$$

where

$P_F(a_1,\ldots,a_M:t)$ is the probability of failure of the valve at time t if amount $a_i$ of the $i^{th}$ type of contamination is present in the valve at time t ($i = 1,2,\ldots,M$),

and

$P(a_1,\ldots,a_M:t)$ is the probability that there will be amounts $a_i$ of the $i^{th}$ type of contamination present in the valve at time t.

In analyzing $P(a_1,\ldots,a_M:t)$, it was assumed that there were N sources of contamination for the valve during its operation: contamination sealed in the certain sites of the system to which the valve was attached. No consideration was given to the analysis of specific sources of contamination before operation of the system, i.e., the environment before the system was sealed, contamination from materials in the system, and so forth. Thus, the analysis of $P(a_1,\ldots,a_M:t)$ was in terms of (a) a probability distribution, $P_i$, representing the initial (after

[8] Tierney, M. S. and Trauth, C. A., Jr., A Model for Determining the Probability of Failure of a Valve Having a Particle Contamination Failure Mode, Sandia Laboratories Research Report, SC-RR-67-772.

sealing) contamination in the $i^{th}$ site and (b) the probability, $\Pi_{iv}^{(j)}(t)$, that a single particle of type j in the $i^{th}$ site, initially, would be found in the valve at time t. These latter probabilities were further analyzed by assuming that certain "transition" probabilities were known (significant factors at the sixth level).

While the authors had no specific system involving a valve in mind, so that the analysis was not carried beyond this level, the approach parallels the general approach presented above, and was not, incidentally, undertaken with the general approach in mind.

The second example of an analysis of a contamination control problem which resembles the general approach presented in this paper is the analysis of the planetary quarantine problem. In planetary quarantine one form of the primary objective may be stated as follows:

> To control contamination so that the probability of biasing life detection and analysis experimentation on a given planet before time T should not exceed $\epsilon$.

In this objective, an "unacceptable penalty" becomes "biasing life detection and analysis experimentation before time T." Unfortunately T must be regarded as unknown. One possible definition of "biasing" in this context is "obtaining results one would otherwise not obtain." This, again, presents some problem since what one would obtain without any contamination is unknown.

An analysis of essentially the above primary objective may be found in a report in which an "unacceptable penalty" was actually left undefined.[9] The ways of incurring unacceptable penalties (biasing experimentation) were assumed to be the missions launched in the vicinity of the planet in question. The missions were divided into "classes" - each class having a possibly different mode of delivering contamination (e.g., landers, flybys and orbiters). Because it was deemed prudent to consider the number of missions in any class unknown (as well as the time period, T, referred to in the primary objective), a simple model of the form found in the general contamination control tree (p.38) could not be used. The model developed was one allowing periodic estimates to be made of the number of missions to be launched in each class. The significant factors influencing the attainment of the primary objective were of the form

> $P_{ik}$ - the probability that a mission of the $i^{th}$ class whose launch is deemed necessary as a result of the $k^{th}$ estimate will "contaminate" the planet in question.

Here, "contaminate" may be understood to mean "deposit contamination in such a way that life detection and analysis experimentation is biased." The exact nature of the model will be found in the report, and need not be elaborated upon here. The secondary objectives arising from the model are of the form

$$P_{ik} \leq \epsilon_{ik} ,$$

[9] Trauth, C. A., Jr., A Sequential Decision Model of Planetary Quarantine Primary Objectives, Sandia Laboratories Research Report, SC-RR-67-462.

where the $\epsilon_{ik}$ can be directly related to the $\epsilon$ appearing in the primary objective.

A complete analysis leading to tertiary objectives has yet to be performed; however, enough is known to show that one of the significant factors appearing at approximately the third level is of the form

$P_L\{n(t) = k\}$ - the probability that the bioburden of a mission at launch is equal to k (for k = 0,1, 2,...).

To attempt to analyze this probability distribution, a model has been developed which may be used to predict microbial survival (or death)[10] in thermal environments. The significant factors appearing in the model include

- The temperature of the thermal environment,
- Time the capsule is exposed to the thermal environment, and
- The number of micro-organisms on the capsule just before its exposure to the thermal environment (or an initial distribution, if more appropriate.).

A first attempt has been made to analyze this last factor in terms of certain assembly contamination parameters,[11] and further work is underway to analyze these parameters in terms of environmental parameters in the hope of obtaining some actionable variables.

Thus, in planetary quarantine, the primary objective has a form like the general contamination control objective. The secondary objectives relate to ways of incurring penalties and are similar in form to those stated earlier in the general context of contamination control. Third level objectives which are known at this time are related to quantities or amounts of micro-organisms present on missions, and those fourth level objectives currently being investigated relate to sources of contamination (assembly, manufacture, etc.). Thus, again, planetary quarantine is another problem area which insofar as it has been analyzed, parallels the general analysis presented earlier in this section. Admittedly, some portions of the hierarchy will differ from the general abstract case presented earlier, but basically, planetary quarantine follows this pattern.

---

[10] Brannen, J. P., A Rational Model for Spacecraft Sterilization Requirements, Sandia Laboratories Research Report, SC-RR-67-256.
_____, "A Rational Model for Thermal Sterilization of Micro-organisms," Mathematical Biosciences, to appear.
_____, "On Logarithmic Extrapolation of Microbial Survivor Curves for Planetary Quarantine Requirements," Journal of Space Life Sciences, to appear.

[11] Sherry, E. J. and Trauth, C. A. Jr., An Assembly Contamination Model, Sandia Laboratories Research Report, SC-RR-66-421.

Incidentally, the "left branches" of the general tree developed on on page does not yield only measurable variables for planetary quarantine. The actual consequences of contaminating a planet cannot be known a priori, so that many of the variables occurring in these branches will present decision situations. Analysis of these left branches is currently underway.[12]

## Conclusions and Comments

The intent of this paper was to develop a theory of contamination control with the following properties. It should be

(1) broad enough to encompass all contamination control problems,

(2) capable of aiding in the formulation of specific contamination control objectives in any given situation,

(3) helpful in determining the activities needed for the achievement of contamination control objectives on some "optimal" utility-penalty basis.

The desirability of such a theory may be attributed to two things. First, the "universality" of contamination control problems, and second, the similar abstract character of many contamination control problems. These lead, inevitably, to the conclusion that there will be many possible ways of solving contamination control problems. The potential gains associated with making a "best" choice from among alternative means of resolving a given problem seem great, so that a theory having the above named attributes seems desirable.

The "systems philosophy" was introduced as an approach to problem resolution primarily concerned with determining activities which "best" achieve some given set of objectives. As a philosophy, it is in many ways analogous to the "scientific method," and is, therefore, not a specific problem resolution scheme but, rather, a point of view. To actually implement the systems philosophy, a framework in which primary objectives (original problem objectives) are linked to "actionable" objectives (directly achievable objectives) was developed. This framework was termed an "objective hierarchy," and the elements occurring in it may be directly related to those occurring in the "systems philosophy." The objective hierarchy appears to be a framework possessing properties (1), (2) and (3), listed above, provided that primary objectives for contamination control are known.

Finally, primary objectives for contamination control were stated, and a partial abstract analysis of these in the objective hierarchy framework was carried out. To examine the validity of this approach, two rather dissimilar specific contamination control problems were presented. Each seemed to be capable of formulation within the framework

---

[12] This research was evidenced by the presentations of N. H. Horowitz and R. W. Davies at the meeting of the Spacecraft Sterilization Advisory Committee of the American Institute of Biological Sciences held in Los Angeles June 19-21, 1967. These gentlemen were addressing themselves to the questions of the nature of contamination and the penalties that might be incurred for its existence in certain spacecraft components. Not unrelated, the article by Horowitz, Davies and R. W. Sharp entitled "Planetary Contamination I: The Problem and the Agreements," Science, Vol. 155, No. 3769, pp. 1501-1505.

developed in this paper, allowing for some variations in specific parameters because of the specific nature of the problems.

While the general approach to contamination control presented in this paper seems applicable to most contamination control situations, it must be understood that it is not a tested theory. To the author's knowledge, no problem in contamination control has been analyzed in this fashion to a complete set of actionable objectives. Thus it is a matter of believing that this can be done. Since a complete set of actionable variables has not been obtained in any contamination control situation, it follows that the theory is equally untried in its ability to obtain "best" means of problem resolution. Nevertheless, with this reservation (and others mentioned below) the approach to contamination control developed in this paper seems, in theory, to satisfy the goals set forth in this paper.

Aside from the untested nature of the approach presented here, there are at least two other reservations about the approach that should be mentioned. The first is that the "true" nature of resource allocation for control has not been thoroughly treated. The method outlined in this paper is an approximate method: a more "realistic" method requires the solution of a rather complex resource allocation problem constrained by the results of solving a number of resource scheduling problems. This occurs when the control of terminal or actionable variables is not independent, i.e., when the method of control of one may influence the control of another. For example, if a clean room is needed for the achievement of one actionable objective and a less costly cleaning facility is needed for the achievement of another, it may be possible, schedule permitting, to use the clean room (which one must have) to replace the cleaning facility, thereby reducing the total resource expenditure. Thus, a need for the "usual" tools of planning is evidenced in the approach presented in this paper. The solution of allocation and scheduling problems is, at times, very difficult,[13] so that the complete success of the approach to contamination control presented here may depend upon one's ability to resolve other difficult problems. The second reservation about the approach outlined in this paper is that it does not consider the organizational and educational aspects of planning. The organizational role that contamination control groups play, the number of groups involved, and the way in which they are coordinated must influence the effectiveness of contamination control actions as well as have some effect on the penalties associated with contamination control activities. Educational factors will influence contamination control "acceptance," effectiveness, activities and so forth. For example, only when the engineering community accepts contamination control as a vital consideration in product design will the potential gains in this area be realized.

Both the educational and organizational aspects of contamination control planning tend to be associated with the implementation of a technical plan derived, possibly, within a framework similar to that presented here. Their importance may be secondary in any relatively short time frame, but, long term, their consideration may well be necessary, and certainly desirable.

---

[13]Pierce, J. F., Jr., Some Large-Scale Production Scheduling Problems in the Paper Industry, Prentice-Hall, Englewood Cliffs, N. J., 1964.

# CLOSING

# CLOSING

## CLOSING OBSERVATIONS

by

H. D. SIVINSKI

Chairman of Symposium

I want to say that for me this has been a good symposium, and probably for a very different reason than for most of the other participants. It has been good for me because I am somewhat new to contamination control, and am therefore on the steep slope of the learning curve. This symposium has taught me much very quickly.

Two things, I think, need further development as a result of this symposium. I am not yet sure how well we did initially on either. The first is, "What really are the problems in the field?" And by the "field" I mean the people who have product to turn out everyday at minimum penalty. We never had a chance to develop very fully this first theme because individual problems multiply so rapidly that such an approach becomes intractable very quickly. So perhaps we dealt with it in the best way, so that one can by inference and by talking to the people at the symposium determine in general what these problems are. The reason one wants to know, of course, is that we have representation here from the instrument manufacturers and the research institutes, and these are the people who can best start to fulfill the needs of the field.

I would caution you, however not to get trapped by becoming so enamored and involved in the solution of a problem that you forget what the problem was all about. Anything that is too complex to use is valueless. Anything that has not been finished when it was needed, and, therefore, is not timely, is valueless. So, in applied research, somebody has to remember what we are out to try to do--and then do that.

The second thing is, "What are the instrumentation manufacturers and research institute people doing and what should they be doing?" The exponential advances in technology are making some problems more tractable every day. The problems that were impossible yesterday are possible today and may be easy to solve tomorrow. My advice is, "Don't sit on your problem; communicate!" Communications, it appears to me, is the great problem in contamination control and instrumentation. I have heard people say, "I solved the problem he's talking about a year ago," or "I had an instrument that did what he is doing a year ago." I think we tend to play our cards too close to our vest.

Now I would like to be philosophical for a moment, and again on the basis of not too long an involvement and therefore more from the position of an outsider looking in. Perhaps this gives me a different view. I will not say it is better, but it may be different from that of you who have dedicated larger portions of your professional life to this field. Contamination control, it appears to me, should have, by now,

grown up to be a recognized consideration in the total product procurement process, through concept, design, manufacture, storage, and use. Based on what I have frequently seen so far, I see no reason for rejoicing at our progress on this point. Contamination control it appears to me, is not properly supported, and I think that we, as a group, should begin to lose patience with ourselves and with any management and engineering and manufacturing segments of the population that cannot or will not stop the waste of the precious commodities of time, effort, and money by refusing to invest properly in the area of contamination control. We ought to start thinking about contamination control a lot sooner in the total process, and it is up to us to educate whoever is blocking that kind of activity. We need to become more aggressive in providing mature consideration of contamination control, and communication with each other is certainly the best way to begin to do that.

# SUMMATION

by

F. J. BEYERLE

Vice Chairman of Symposium

This symposium has shown that automation and instrumentation for the measurement of contamination with any degree of reliability and reproducibility can still be considered in its embryonic stage for NASA aerospace applications.

For example, we are continually searching for automatic methods of analyzing organic compounds for nonvolatile residue content. We have not found one. We need an instrument that can reliably determine particulate contamination in liquid systems on a high volume or continous flow basis. We have not found one. We need an instrument to detect contamination in gaseous systems, such as $N_2$, $O_2$, He, or gas bearing systems. We have not found one. Finally, we have not fully accepted automatic particle counters for use in aerospace clean rooms as production models.

This means that we still have a long way to go to attain these goals, and, hopefully, we in NASA are looking forward to help resolve these problems. We recognize, also, the complexities involved in meeting the many faceted requirements in the NASA contamination control programs which may create problems for manufacturers of instrumentation.

Finally, as the NASA representative for this symposium, I want to thank the AEC, Sandia Corporation, and especially Mr. Sivinski and his staff for the fine job they have done in making this symposium a smooth running performance.

DISTRIBUTION:

United States Atomic Energy
Commission, Albuquerque
Operations Office (2)
S. A. Upson
Attn: M. Roepke

United States Atomic Energy
Commission, Division of
Technical Information (3)
P. O. Box 62
Oak Ridge, Tennessee

United States Atomic Energy
Commission, Declassification
Branch, P. O. Box E
Oak Ridge, Tennessee

United States Atomic Energy
Commission, Sandia Area Office
P. O. Box 5400 (2)
Albuquerque, New Mexico, 87115
Attn: K. R. Braziel

United States Atomic Energy
Commission, Washington, D. C.,
20545 - Attn: H. Gilbert

Aerojet General Corp., MSC/WSTF
P. O. Box MM
Las Cruces, New Mexico 88001
Attn: W. E. Mountain

Air Tech
2108 Carterdale
Baltimore, Maryland 21209
Attn: S. B. Steinberg

Ames Laboratory
Iowa State University
Ames, Iowa 50010
Attn: M. D. Voss

Barnstead Still and Sterilizer Co.
2 Lanesville Terrace,
Boston, Mass. 02131
Attn: V. C. Smith

Battelle Memorial Institute (2)
505 King Avenue
Columbus, Ohio 43201
Attn: J. M. Pilcher
J. A. Gieseke

Bausch & Lomb, Inc.
635 St. Paul St.
Rochester, New York, N. Y.
14602
Attn: A. E. Martens

Bell Telephone Laboratories
Room 4-B-629
Holmdel, New Jersey 07733
Attn: T. F. Egan

Bell Telephone Laboratories, Inc. (3)
Murray Hill, New Jersey 07971
Attn: R. Brown
H. M. Cleveland
G. E. Helmke

Bendix Corporation
Kansas City, Missouri 64113
Attn: R. T. Foster

The Bendix Corporation
Kansas City Division
P. O. Box 1159
Kansas City, Missouri 64141
Attn: R. B. Hedrick

Bendix (3)
Launch Support Division
P. O. Box 21086
Kennedy Space Center
Florida 32815
Attn: O. M. Briles
J. Killion
W. F. Underwood

The Boeing Company
4667 E. View Drive
New Orleans, Louisiana 70126
Attn: G. P. Schonhard

Cambridge Filter Corporation
P. O. Box 1255
Syracuse, New York, 13201
Attn: R. T. Goulet

Chrysler Corporation, Space
Division, P. O. Box 29200
New Orleans, Louisiana 70129
Attn: N. E. Hildebrandt

Consultant-Sandia
4960 Iroguois, Apt #2
San Diego, California 92117
Attn: J. A. Paulhamus

Rep-Contamination Control Journal
1001 Silver, SE
Albuquerque, New Mexico 87106
Attn: F. Kutnewsky

Coronado Building
Santa Fe, New Mexico 87501
Attn: E. O. Goodrich, Jr.

Envirco
630 Haines, N.W.
Albuquerque, New Mexico
Attn: R. C. Marsh

Fairchild Hiller Corporation
Fairchild Drive
Germantown, Maryland 20767
Attn: A. P. Ross

DISTRIBUTION: (cont)

Farr Company
2301 E. Rosecrans
El Segundo, California 90009
Attn: P. A. Labadie

Flanders Filters, Inc.
Pleasure Drive
Riverhead, New York 11901
Attn: G. H. Cadwell

General Electric
Daytona Beach, Florida 32015
Attn: D. L. Conley

General Electric Co. (2)
P. O. Box 8555
Philadelphia, Pa. 19101
Attn: Martin G. Koesteres
Western Electric

J. R. Gillis

General Electric
455 Clearview St.
Pottstown, Pa. 19464
Attn: J. J. Gergle

General Electric Co.
Room M-2101, P. O. Box 8555
Phildelphia, Pa. 19101
Attn: E. Fried

Grumman Aircraft Engineering
Corp. (3)
Bethpage, New York 11714
Attn: L. J. Kolodzeski
D. S. Marinaro
H. J. Marx

Grumman Aircraft Engineering
Corp., MSC/WSTF
White Sands, Las Cruces,
New Mexico 88001
Attn: J. A. Hicks

Grumman Aircraft Engineering
Corp., 2250 Missouri
Las Cruces, New Mexico 88001
Attn: B. G. Seib

Hayes International Corp.
P. O. Box 2287
Birmingham, Alabama, 35201
Attn: (Mrs.) J. Y. Ellenburg

Heating, Piping and Air Conditioning, 10 S. LaSalle St.
Chicago, Illinois 60603
Attn: T. C. Elliott

High Accuracy Products Corp.
141 Spring St.
Claremont, California 91711
Attn: L. D. Carver

IIT Research Institute (3)
10 West 35th St.
Chicago, Illinois 60616
Attn: M. R. Jackson
A. Lieberman
Z. P. Saperstein

Jet Electronics Corp. (3)
4400 Silver, SE
Albuquerque, New Mexico 87108
Attn: G. R. Birrell
D. W. Smith
J. E. Tillman

Jet Propulsion Laboratory
4800 Oak Grove Drive
Pasadena, California 91103
Attn: G. L. Hobby

Killsman Instrument
Underhill Boulevard
Syosset, Long Island, New York
Attn: P. I. Ravitz

Lawrence Radiation Laboratory
Technical Information Division
Livermore, California

Los Alamos Scientific Laboratory
P. O. Box 1663
Los Alamos, New Mexico 87544
Attn: D. D. Meyer, H-1

Lovelace Foundation,
5200 Gibson Blvd., S. E.
Albuquerque, New Mexico 87108
Attn: A. H. Schwichtenberg

Lovelace Clinic
5200 Gibson Blvd., S. E.
Albuquerque, New Mexico
Attn: J. G. Whitcomb

Marshall Space Flight Center (25)
2811 Irwin Rd. S. W.
Huntsville, Alabama 35801
Attn: F. J. Beyerle

Mason and Hanger-Silas Mason Co.
P. O. Box 647
Amarillo, Texas 79105
Attn: G. H. Burling

Mason and Hanger-Silas Mason Co.
P. O. Box 561
Burlington, Iowa 52601
Attn: J. R. Polson

DISTRIBUTION: (cont)

Martin Marietta (2)
P. O. Box 179
Denver, Colorado 80201
Attn: J. P. McDonald
M. Piccone

Martin Marietta Corp.
P. O. Box 2371
Denver, Colorado 80201
Attn: Milt W. McKenzie

Martin Marietta
Waterton Plant
Denver, Colorado
Attn: J. E. Cole

Mines Safety Appliance
1321 W. 12th
Los Angeles, California 90291
Attn: J. W. Becker

Mine Safety Appliances
201 N. Braddock Ave.
Pittsburgh, Pa. 15208
Attn: H. C. Elinsky

NASA
Marshall Space Flight Center
R-P & VE-VNR-BEC
Huntsville, Alabama 35810
Attn: C. M. Rush

NASA
Marshall Space Flight Center
Huntsville, Alabama 35812
Attn: D. N. Vickers

NASA
Ames Research Center
Moffett Field, California 94035
Attn: J. P. Burgard

NASA
John F. Kennedy Space Center (3)
320 Harbor Drive
Cape Kennedy, Florida
Attn: L. C. Hull, Jr.
J. B. Gayle
E. L. Green

NASA
Rt 1, P. O. Box 114
Cocoa, Florida 32922
Attn: W. F. Petty

NASA
Goddard Space Flight Center,
Greenbelt Road
Greenbelt, Maryland
Attn: F. N. LeDoux

NASA
WSTF/MSC
1987 Crescent Drive
Las Cruces, New Mexico 88001
Attn: B. W. Peipelman

NASA
P. O. Drawer MM
Las Cruces, New Mexico 88001
Attn: I. D. Smith

NASA
Manned Space Center
Houston, Texas
Attn: Q. T. Ussery

NASA (2)
400 Maryland Ave. SW
Washington, D. C. 20546
Attn: D. E. Negola
A. H. Neill

National Bureau of Standards
B326 Chemistry Building
Washington, D. C. 20234
Attn: R. A. Paulson

North American Aviation-Autonetics
1963 Green Leaf #C
Anaheim, California 92801
Attn: F. M. Allison

Pall Corporation
Station E, P. O. Box 11161
Albuquerque, New Mexico
Attn: L. O. Poage

U. S. Public Health Service
4402 N. 7th St.
Phoenix, Arizona 85014
Attn: W. W. Bond

U. S. Public Health Service (2)
National Communicable Disease
Center, 4402 N. 7th St.
Phoenix, Arizona 85014
Attn: M. S. Favero
N. J. Petersen

Royco Instruments, Inc. (2)
141 Jefferson Dr.
Menlo Park, California 94025
Attn: H. M. Ogle
J. R. Weersing

Royco Instruments, Inc.
432 31st St.
Manhattan Beach, California
Attn: J. Theis

Space Nuclear Propulsion Office
Lewis Research Center
Cleveland, Ohio
Attn: D. C. Adams

DISTRIBUTION: (cont)

University of New Mexico
Dept of Microbiology
Albuquerque, New Mexico 87106
Attn: C. Franklin

University of New Mexico (2)
Department of Biology
Albuquerque, New Mexico 87106
Attn: J. W. Beakley
L. W. Hughes

Western Electric Company, Inc.
555 Union Blvd.
Allentown, Pa. 18103
Attn: P. W. Morrison

Western Electric Company, Inc.
P. O. Box 900
Carter Road
Princeton, New Jersey 08540
Attn: E. J. Shaw

Western Electric Engineering
Research Center
P. O. Box 900
Princeton, New Jersey 08540
Attn: C. Altman

Western Electric
P. O. Box 241
Reading, Pa. 19603
Attn: R. A. Yeich

F. L. Fey, Jr.
3921 Van Dyke,
White Bear Lake, Minnesota
55110

J. A. Hornbeck, 1
W. J. Howard, 1000
C. F. Bild, 1100
J. E. McDonald, 1110
A. J. Quant, 1113
W. M. O'Neill, 1120
L. M. Berry, 1130
N. J. DeLollis, 1133
C. W. Jennings, 1133
W. Herrmann, 1140
O. L. Burchett, 1144
E. R. Dunaway, 1144
T. R. Guess, 1144
L. A. Hopkins, 1300
G. L. Krieger, 1315
J. H. Findlay, 1400
W. A. Gardner, 1500
A. B. Machen, 1600
R. W. Henderson, 2000
L. J. Heilman, 2100
C. R. Barncord, 2200
L. J. Paddison, 2400
H. E. Lenander, 2500
J. R. Meikle, 2520
J. W. Jones, 2540
D. E. Fjelseth, 2541
D. L. Clement, 2543
E. T. Schreiner, 2545
R. E. Hepplewhite, 2550
J. R. Sublett, 2560
D. W. Ballard, 2564 (5)
L. J. Klamerus, 2564 (3)
J. C. Mashburn, 2564
W. E. Neitzel, 2564
F. W. Oswalt, 2564
D. M. Davis, 2564
C. L. Page, 2565
E. S. Roth, 2565
H. D. Sivinski, 2570 (25)
C. A. Trauth, Jr., 2571
D. M. Garst, 2572
K. F. Lindell, 2572
M. E. Morris, 2572
W. J. Whitfield, 2572
R. B. Powell, 3000
K. A. Smith, 3100
R. F. Utter, 3132
D. S. Tarbox, 3200
S. P. Bliss, 3300
M. K Linn, 3400
N. S. Hey, 3431
C. W. Campbell, 4000
R. G. Luckey, 4100
R. J. Hansen, 4200
C. E. Runyan, 4220
J. A. Kenagy, 4224
C. R. Pritchett, 4300
R. E. Hopper, 4500
S. L. Smith, 4514
C. R. Mills, 4541
H. H. Baxter, 4542
C. E. Champe, 4542
E. R. Gruer, 4542
J. D. Holton, 4542
E. E. Rush, 4542
D. W. Aubuchon, 4543
E. L. Drake, 4543
G. B. Lenert, 4543
R. E. Perryman, 4543
L. C. Roper, 4544
C. J. McGarr, 4600
T. B. Cook, Jr., 5000
R. S. Claassen, 5100
T. B. Cook, Jr., Actg, 5200
L. D. Smith, 5500
M. C. Reynolds, 5530
B. H. VanDomelen, 5530
R. T. Dillon, 5590
E. W. Shepherd, 5590
D. B. Shuster, 5600
C. T. Ross, Jr., 6000
R. A. Bice, 7000
B. F. Murphey, 7100
L. E. Hollingsworth, 7200

DISTRIBUTION: (cont)
L. E. Lamkin, 7300
B. S. Biggs, 8000
L. Gutierrez, 8100
C. H. DeSelm, 8200
G. A. Fowler, 9000
J. H. Scott, 9200
A. Y. Pope, 9300
W. C. Scrivner, 9400
M. Sanguinetti, 3412 (4)
L. C. Baldwin, 3412
B. F. Hefley, 8232 (4)
C. H. Sproul, 3428-2 (10)